

*1946 Book of*  
**A.S.T.M. STANDARDS**

*Including*  
**TENTATIVES**  
(A Triennial Publication)



Reg. U. S. Pat. Off.

**PART III-B**  
**Nonmetallic Materials—**  
Electrical Insulation, Plastics, Rubber, Paper,  
Shipping Containers, Adhesives

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## FOREWORD

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The Book of A.S.T.M. Standards, of which this is the 1946 edition, is normally a triennial<sup>1</sup> publication, with Supplements issued in the intervening years. It contains the formally adopted A.S.T.M. Standards and the A.S.T.M. Tentative Specifications, Methods of Test, and Definitions.

Prior to this edition, the Book of Standards was issued in three parts. Today, with the extension of Society work into many new fields and the great stimulus given by war activities to the development of tests and specifications for materials, the 1946 edition of the Book of Standards comprises about 7000 pages. To continue to publish this amount of material in three parts would result in books too large for convenient use and too bulky and heavy for binding, except at prohibitive cost. Hence it was decided to publish the 1946 Book of Standards in five parts, as follows:

*Part I-A—Ferrous Metals* (Steel, Wrought Iron, Cast Iron, Magnetic Properties, Malleable-Iron Castings, Ferro-Alloys, Iron-Chromium-Nickel and Related Alloys).

*Part I-B—Non-Ferrous Metals* (Copper and Copper-Alloy Wires, Non-Ferrous Metals and Alloys, Light Metals, Electrodeposited Metallic Coatings, Metal Powders and Metal Powder Products, Electrical-Heating Alloys, Copper and Copper Alloys, and Die Cast Metals and Alloys).

*Part II—Nonmetallic Materials—Constructional* (Cement, Lime, Gypsum, Masonry Units, Natural Building Stone, Mortar for Unit Masonry, Thermal Insulating Materials, Refractories, Glass and Glass Products, Clay Pipe, Concrete Pipe, Drain Tile, Concrete and Concrete Aggregates, Road and Paving Materials, Soils, Waterproofing and Roofing Materials, Wood and Wood Preservatives, Paint, Varnish, Lacquer, and Related Products, Naval Stores, Fire Tests, Thermometers).

*Part III-A—Nonmetallic Materials* (Coal and Coke, Gaseous Fuels, Petroleum Products, Aromatic Hydrocarbons, Soaps, Water, Textiles, Thermometers).

*Part III-B—Nonmetallic Materials* (Electrical Insulating Materials, Plastics, Rubber, Paper, Shipping Containers, Adhesives, Thermometers).

In these five parts are included the 1330 A.S.T.M. Standards and Tentatives in effect at the time of publication, exclusive of the 35 A.S.T.M. Methods of Chemical Analysis of Metals which appear in a separate publication bearing that title.

The annual Supplements to each part issued in the succeeding years will contain the newly adopted or revised Standards and new or revised Tentatives. These Supplements should be consulted for changes in status and for current revisions of Standards and Tentatives. The combined Index to A.S.T.M. Standards, issued annually, will also be helpful in this connection and in locating any desired standard.

<sup>1</sup>The present edition is being issued with only a two-year interval since the preceding edition, due to the early exhaustion of the supply of the book.

*Standards* appear in the front of each part, and comprise those specifications and methods of test that have been formally adopted by the Society, requiring a letter ballot approval by the entire membership.

*Tentatives*, which appear in the back of each part, have been approved by the sponsoring committee as representing the latest thoughts and practices and have been accepted by the Society in accordance with established procedures for use pending adoption as standard.

In accordance with Society regulations, standards that have been published for a period of six years or more without revision have been reviewed by the responsible standing committees prior to the appearance of the Book and have been reapproved in their present form.

The appearance of this Book is a tribute to the sincere efforts of the technical committees responsible for the development and review of the standards, and of the many individuals serving on the committees. The Society's time-tested standardization procedure provides for adequate representation on technical committees of producers and consumers and those having a general interest in the material or subject in question. Opportunity is provided during the period of promulgation for full consideration by all interested parties through publication as tentative before adoption as standard. As developments and advances take place, requirements are suitably revised.

## GUIDE TO THE USE OF THE BOOK OF STANDARDS

**T**HE standards in this Book are assembled in a sequence determined by the specific materials or products to which they apply. The Table of Contents in each part is presented in duplicate—one a list of standards in the order in which they appear, the other a list of the standards in numeric sequence of their serial designations. In addition, a separate listing (printed on green colored stock) is given of the tentative specifications and methods of test just preceding that portion of the book beginning on p. 495.

A subject index of the standards and tentatives in this part appears at the back of this volume (see page 1327). This index should be the most convenient means of locating any standard appearing in the volume, especially if only the general subject matter covered is known.

A separate combined Index, covering all standards and tentatives in Parts I-A and I-B, II, and III-A and III-B of the Book of A.S.T.M. Standards, in the annual Supplements thereto, and in the book of A.S.T.M. Methods of Chemical Analysis of Metals, is also issued annually, which should be of convenience to all users of the Book of Standards. The combined Index will be revised annually to incorporate the latest references to the standards, whether they appear in the Book of Standards or in the 1947 or 1948 Supplements. This Index should be of convenience to all users of the Book of A.S.T.M. Standards and its Supplements.

A numeric list with complete titles of the current standards and tentatives also appears with this Index.

Comments or suggestions intended to improve the utility of the Book of Standards will be welcome.



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### Nonmetallic Materials—Electrical Insulation, Plastics, Rubber, Paper, Shipping Containers, Adhesives<sup>1</sup>

(For standards covering metals, see Parts I-A and I-B; for nonmetallic materials—constructional, see Part II; and for standards covering fuels, petroleum, aromatic hydrocarbons, soaps, water, and textiles, see Part III-A)

In the serial designations prefixed to the following titles, the initial letter, indicative of the general classification, and the first number are permanent. The number following the dash indicates the year of original issue as tentative or of adoption as standard, or, in the case of revision, the year of last revision. Thus, standards adopted or revised during the year 1946 have as their final number, 46. Tentatives are identified by the letter T.

In the following listing, the Standards and Tentatives are grouped according to materials to which they apply. Listings of Tentatives appear in italics.

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The Emergency Standards Procedure was discontinued by the Society in 1946. Accordingly, the Emergency Standards have either been issued as tentatives or discontinued, and in the former case appropriate reference is made in explanatory footnotes appearing elsewhere in these Contents. A number of the Emergency Alternate Provisions have resulted in revisions in the standards and tentatives, while all the others have been discontinued.

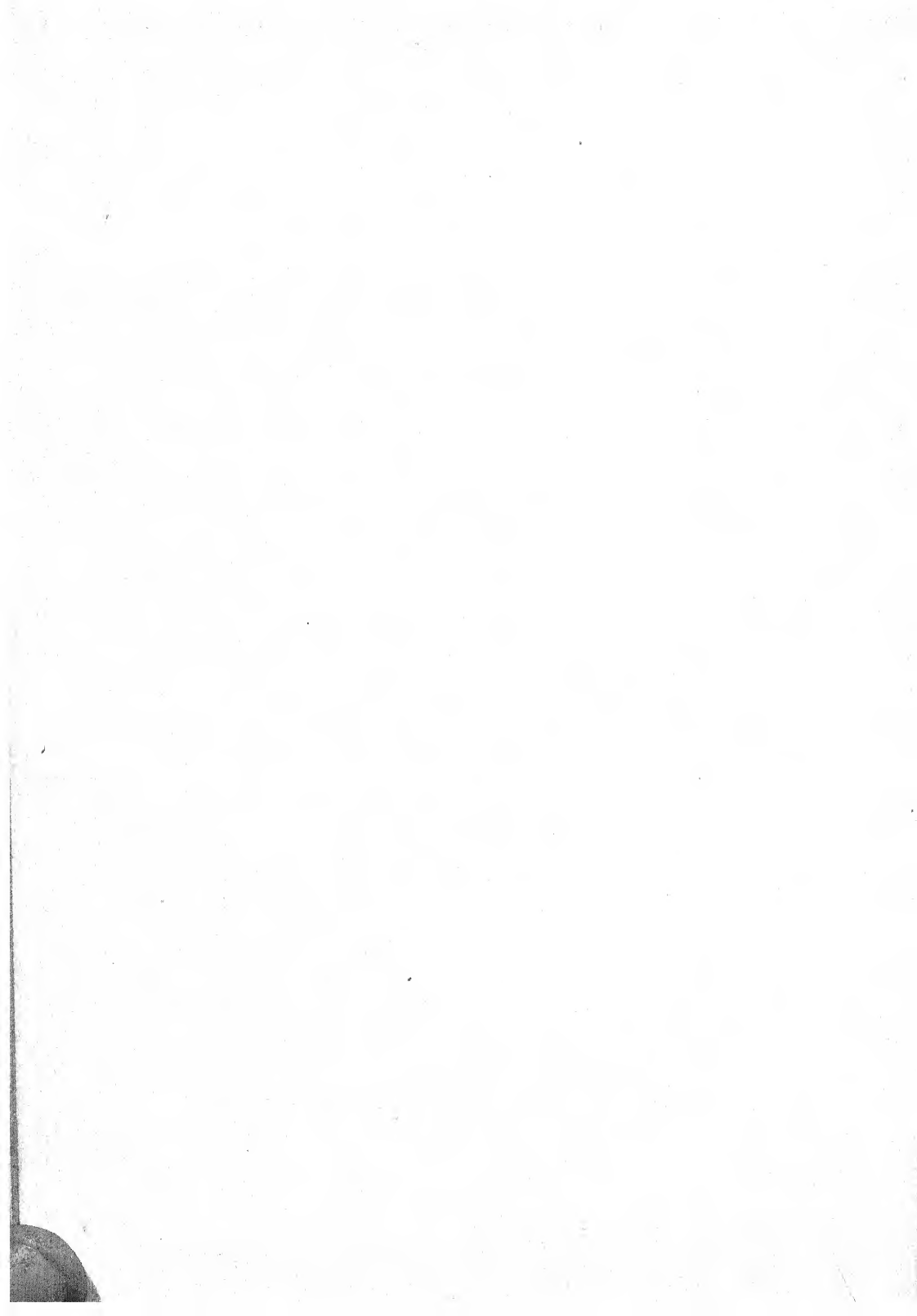
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This list in numeric sequence includes only those standards and tentatives appearing in this book. For a complete list in numeric sequence of all standards and tentatives issued by the Society, see the current edition of the combined Index to A.S.T.M. Standards which is published separately and furnished with this volume. The list in the combined index will be particularly helpful after the appearance of the 1947 and 1948 Supplements, since it will contain references to the new and revised standards and tentatives appearing in the 1947 and 1948 Supplements as well as to those current standards and tentatives appearing in this book.

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† A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1323.

<sup>1</sup> This is in effect a tentative revision of, and, when adopted, is to replace Standard Methods D 117 - 43.

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\* Approved as American Standard by the American Standards Association.

† A "tentative revision" of this standard appears in the Tentative Revisions of Standards section at the back of this book, see p. 1323.

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<sup>2</sup> These specifications were formerly published as Emergency Specifications ES - 6a.

<sup>3</sup> These specifications were formerly published as Emergency Specifications ES - 41.

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#### EMERGENCY STANDARDS AND EMERGENCY ALTERNATE PROVISIONS

The Emergency Standards Procedure was discontinued by the Society in 1946. Accordingly, the Emergency Standards have either been issued as tentatives or discontinued, and in the former case appropriate reference is made in explanatory footnotes appearing elsewhere in these Contents. A number of the Emergency Alternate Provisions have resulted in revisions in the standards and tentatives, while all the others have been discontinued.

\* Approved as American Standard by the American Standards Association.

\* This is in effect a tentative revision of, and, when adopted, is to be added to Standard Specifications E 1 - 46.

# Standard Methods of TESTING SHELLAC USED FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 411 - 44

ADOPTED, 1940; REVISED, 1942, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 411; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods cover tests for shellac in the dry or powdered form to be used for electrical insulating purposes.

## General Tests

2. Each of the following tests shall be made in accordance with the procedures described in the Standard Methods of Sampling and Analysis of Shellac (A.S.T.M. Designation: D 29) of the American Society for Testing Materials.<sup>3</sup>

- (a) Sampling,
- (b) Insoluble matter,
- (c) Iodine number,
- (d) Moisture content,
- (e) Wax,
- (f) Ash, and
- (g) Orpiment.

## POLYMERIZATION TIME<sup>4</sup>

### Apparatus

3. The apparatus shall consist of the following:

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1935 to 1940, being revised in 1937.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part II.

<sup>4</sup> This property is also known as "life under heat," or "life of the resin."

(a) *Test Tubes*.—Two  $\frac{3}{4}$ -in. glass<sup>5</sup> test tubes, supported by a wire rack to a depth of 4 in. when placed in an oil bath. It is essential that the test tubes be supported and maintained in a vertical position. The rack shall be so constructed as to permit free circulation of oil around the test tubes.

(b) *Oil Bath*.—An oil bath of such construction as to permit of maintaining a uniform test temperature within 1 C. It is essential to use a mechanical stirring device in the oil bath to maintain a uniformly distributed temperature. The oil used shall be a mineral oil having a Saybolt Universal viscosity of approximately 150 sec. at 100 C.

(c) *Burner*.—A bunsen burner for heating the oil bath.

(d) *Glass Rod*.—A smooth glass rod 5 mm. in diameter. A small mark or indicator shall be placed on top of the rod or the glass formed-over to facilitate readings as outlined in Section 6 (d). The sides of the glass rod, near the end which is immersed in the shellac, shall be flattened slightly.

(e) *Thermometer*.—A suitable ther-

<sup>5</sup> Pyrex or quartz glass is satisfactory for this purpose.

mmometer to indicate the test temperature.

#### Conditioning of Sample

4. (a) Samples of shellac before testing shall be rolled and mixed well on clean paper and then carefully dried as described in Paragraph (b).

(b) A flat-bottomed dish, about 4 in. in diameter containing a 5-g. sample of shellac, shall be placed in a well-ventilated gas or electric oven at a temperature of  $41 \pm 2$  C. for approximately 16 hr. (or overnight). It is essential to have the sample spread out on the dish during the drying period.

(c) After drying, the sample shall be transferred immediately to a clean, dry bottle, stoppered tightly, and allowed to cool in the bottle. The bottle shall not be opened except when a specimen is being removed for test.

#### Test Specimens

5. (a) Each test specimen shall consist of 2 g. of the shellac sample taken from the stoppered bottle.

(b) Two specimens of each shellac sample shall be tested.

#### Procedure

6. (a) A specimen of shellac shall be transferred from the stoppered bottle to the  $\frac{3}{4}$ -in. glass test tube. The tube shall then be inserted and held securely in a vertical position in the test rack placed in the oil bath maintained throughout the test at the specified test temperature within plus or minus 1 C.

NOTE.—The method is suitable for testing at any temperature between 125 and 225 C.

(b) The time when the test tube enters the oil bath shall be recorded.

(c) Using the 5-mm. glass rod, the specimen shall be stirred gently for the first 3 min. of the test so that the shellac becomes well melted in as short a time as possible. It is essential that there be no stirring after the first 3-min. period.

(d) At the end of each minute thereafter the glass rod shall be given a slight twist (turning approximately 90 deg.). In the early stages of the test, and before the polymerization point is reached, the glass rod will remain in the position to which it has been turned. When, however, the shellac takes on a rubbery set, there will be a definite turning or twisting back of the rod following the 90-deg. twist. This is the end point. (In the early stages of the test there may be a tendency for the glass rod to move back slowly after twisting with the fingers. This slight movement should not be confused with the definite "twist-back" observed when the end point is reached.)

(e) The temperature shall be recorded each minute during the test. The average of all these readings shall be considered the test temperature.

#### Polymerization Time

7. The elapsed time in minutes, from the time of entry of the specimen into the bath and including the 3-min. stirring period, until the first "twist-back" of the glass rod is noted, shall be recorded as the "polymerization time."

#### Report

8. The report shall include the following:

- (1) The polymerization time to the nearest minute for each specimen,
- (2) The average of the values in Item (1) above, and
- (3) The test temperature.

#### FLOW TEST

##### Purpose

9. (a) The purpose of this test is to determine the flow of shellac when subjected to a definite temperature under the conditions of test indicated.

(b) The method consists in melting a sample of ground shellac in a graduated test tube and then tilting the tube to a definite angle while maintained at a

stated temperature in order to permit the shellac to flow down the tube.

(c) Two methods are provided, as follows:

*Method A.*—In this method the time required for the shellac to flow specified distances along the test tube is observed.

*Method B.*—In this method the total distance the shellac flows along the test tube in a specified time is observed.

#### Apparatus

10. (a) *Method A.*—A suitable form of testing apparatus for use in method A is shown and described in the Appendix.

(b) *Method B.*—A suitable form of testing apparatus for use in method B is shown and described in the Appendix. Any apparatus that will provide for accurately maintaining the required test temperature and the required positions of the test tubes may be used in method B.

#### Test Specimens

11. (a) Each test specimen shall consist of 2 g. of shellac, coarsely ground (approximately to pass a No. 20 sieve). The specimens shall be spread out in a shallow vessel and placed in a desiccator over a saturated solution of sodium dichromate with an excess of solid salt and left in this atmosphere (52 per cent relative humidity) at room temperature for at least 24 hr. The specimens shall be tested immediately upon removal from the desiccator.

(b) Two specimens shall be tested.

#### Preparation of Apparatus

12. (a) The two specimens of shellac shall be placed in separate glass test tubes, care being taken that the specimen in each tube is at the bottom and that none of the powdered shellac adheres to the walls of the glass tube.

The top level of the dry shellac in each tube shall be read on the millimeter graduated scale. The tubes containing the specimens shall then be clamped in place in the testing fixture (see Section 10; also Fig. 1 in the Appendix).

(b) The testing fixture with the glass test tubes in a vertical position shall be inserted in an oil bath maintained at the desired test temperature within plus or minus 1 C.

NOTE.—The method is suitable for testing at any temperature between 100 and 125 C.

(c) The top surface of the specimen in each tube should be level and at right angles to the walls of the tube.

(d) The specimens shall be allowed to melt for 3 min.

#### Method A

#### Procedure

13. At the end of the 3-min. melting period, the test tubes shall each be placed at an angle of 15 deg. from the horizontal, with the corked ends down (see Section 10 (a); also Fig. 2 in the Appendix), and with the breather tube extending above the level of the oil bath. The change from the vertical position to the flow position shall be made as quickly as possible. With the oil bath maintained at the desired test temperature, the total time required for the shellac in each tube to flow from the initial level of the shellac to each centimeter marking along the tube shall be recorded. The test in each tube shall be discontinued when the flow is 9 cm. or the total time is 20 min.

#### Report

14. The report of tests by method A shall include the following:

(1) The time required for each centimeter distance of flow for each specimen,

(2) A curve showing the data reported in Item (1) above, with time plotted as

abscissas and flow in centimeters as ordinates,

(3) The test temperature,

(4) The angle of the test tubes during the flowing period, and

(5) The atmospheric temperature and humidity of the laboratory.

### Method B

#### Procedure

15. (a) At the end of the 3-min. melting period, the test tubes shall each be placed at an angle of 15 deg. from the horizontal, with the corked ends down (see Section 10 (b); also Fig. 2 in the Appendix), and if a breather tube is used it shall extend above the level of the oil bath. The change from the vertical position to the flow position shall be made as quickly as possible. With the oil bath maintained at the desired test temperature, the test tubes shall be allowed to remain in the bath in this position for exactly 12 min.

(b) The test tubes shall then immedi-

ately be removed, placed in a vertical position, cooled, wiped, and the flow of the shellac in each tube measured by reading the distance between the initial point (Paragraph (a)) and the end of the flow tongue. The "feather" at the very tip of the tongue should be disregarded.

NOTE.—This "feather," caused by separation of wax from the shellac, is more noticeable with some shellacs than with others. It can readily be distinguished from the main body of shellac as it is always of different color.

#### Report

16. The report of tests by method B shall include the following:

(1) The flow expressed in millimeters for each specimen,

(2) The average of the values in Item (1) above,

(3) The test temperature,

(4) The angle of the test tube during the flowing period, and

(5) The atmospheric temperature and humidity of the laboratory.

## APPENDIX

### DESCRIPTION OF APPARATUS FOR DETERMINING THE FLOW OF SHELLAC

The apparatus shown in Figs. 1 and 2 consist of the following:

(a) *Test Tubes*.—Two test tubes, *A*, of the following types should be used:

(1) For use with method A, the tubes should be preferably a red-lined, heat-resistant glass, 13 cm. in length, 2.5 cm. in outside diameter, and 1.5 mm. in wall thickness. They should be graduated in 5-mm. divisions beginning 1.1 cm. ( $\frac{1}{8}$  in.) from the outside bottom and extending upward to 100 mm. Every 10-mm. line should be numbered. These tubes are to be used for holding the specimen *S*.

(2) For use with method B, plain heat-resistant glass tubes 12.5 cm. in length and 2.5 cm. in outside diameter.

The test tubes should be stoppered with tightly fitting corks through which extend small breather tubes, *B*.

(b) *Support*.—A fixture *C* for holding the glass tubes in the correct position. This consists of two disks of brass supported as a pen-

dulum as indicated. The disks are free to turn on the supporting shaft and are held in the desired position by the pin *D*. The tubes are supported on narrow V-blocks, and held in position by coil springs *H*, attached to disks *C*.

(c) *Oil Bath*.—A metal bath *E*, heated by a bunsen burner or an electric immersion heater. Oil having a Saybolt Universal viscosity of approximately 150 sec. at 100 C. is suitable. Glycerol or a clear oil should be used where visual observations during the flowing period are required as in method A. It is essential to use a mechanical stirring device *F*, to maintain a uniformly distributed temperature. The use of a glass tank or container with an immersion heater is required for method A, where it is desired to observe the flow during the test and to record the time for the shellac specimen to flow various stated distances. For method B any suitable container may be used.

(d) *Thermometer*.—A thermometer *G*, to indicate the test temperature. The A.S.T.M. Partial-Immersion Thermometer graduated in

either Centigrade or Fahrenheit degrees as specified, the range being  $-5$  to  $+300$  C. or  $+20$  to  $+580$  F., and conforming to the requirements for thermometer 2C-39 or 2F-39, respectively, as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1)<sup>6</sup> is satisfactory for this purpose.

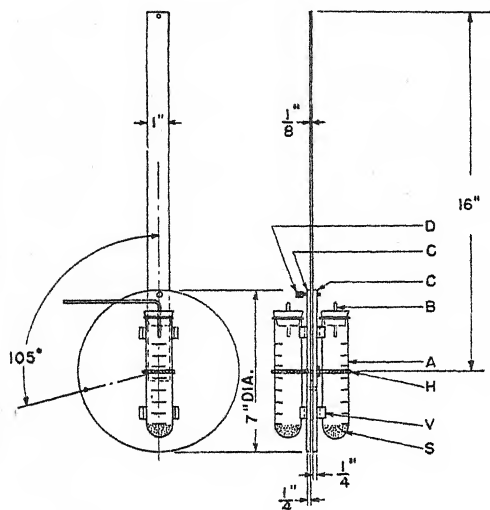


FIG. 1.—Flow Test Fixture Showing Essential Parts with Test Tubes in Vertical Position.

<sup>6</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

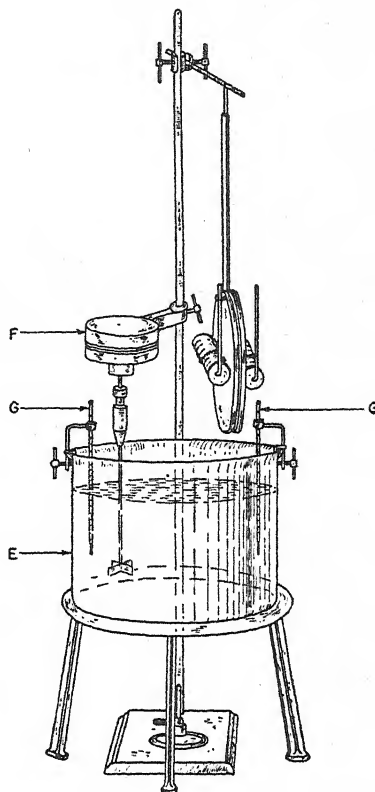


FIG. 2.—Apparatus Arranged with Tubes in Inclined Position for Immersion in Bath for Flow Test.



## *Standard Methods of*

# TESTING MOLDING POWDERS USED IN MANUFACTURING MOLDED ELECTRICAL INSULATORS<sup>1</sup>



A.S.T.M. Designation: D 392 - 38

ADOPTED, 1938.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 392; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover tests for certain physical properties of molding powders, used for the manufacture of electrical insulators and other plastic molded parts. These powders consist of an organic binder of either the thermoplastic or heat-reactive type and organic or inorganic fillers or mixtures of such fillers.

### SAMPLING

#### Scope

2. This sampling procedure is intended for obtaining a representative sample of a batch or shipment of molding compound from the manufacturer's original packages.

#### Procedure

3. (a) If the complete shipment is indicated by the manufacturer's marking to consist of only one batch or "run" of material, the number of packages

selected for sampling shall be not less than 10 per cent of the packages in the batch, and in any case, not less than 3 packages. The packages selected for sampling may be chosen at random, or in case the manufacturer has marked the packages to indicate the order in which they were filled from his batch, the selection may be made with reference to this order of filling, if desired by the purchaser. If the shipment consists of more than one batch or "run" of material, each batch shall be sampled separately to produce one composite blended sample for examination for each batch in the shipment.

(b) The packages selected shall be opened carefully, making sure that no contamination enters them from scale, paint, shattered heads, torn liners, or other causes. From each standard size drum sampled, an equal number of scoopfuls of material shall be taken from a point about 3 in. below the surface. From smaller packages the sample shall be taken from a point 1 in. below the surface. The material shall be transferred to a clean, dry, sheet metal or glass container, capable of

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1934 to 1938, being revised in 1935. Editorially revised in 1941.

being tightly closed. The gross quantity of sample taken shall be approximately twice that estimated to be required for the tests which are to be made. This allowance is made in order to permit repeat tests without re-sampling. The composite sample shall be blended thoroughly before using for the desired tests.

NOTE.—Drums of molding powder in transit, and frequently during packing at the manufacturers' plant, become shaken down and frequently are so densely compacted that only a few ounces of material would fall out if the container were inverted. The few coarse particles not bound in the mass show plainly on the top surface, giving the impression that the fines have sunk and the coarse particles risen. A check-up by sieve analysis of samples taken just under the surface, and throughout the drum, ordinarily fails to disclose segregation.

#### PARTICLE SIZE DETERMINATION OR SIEVE ANALYSIS

##### Scope

4. Two procedures, Methods *A* and *B*, are given for determining the size distribution of the particles in the molding powder being examined. Method *A* is a complete sieve analysis and Method *B* a shorter check method by which the relative quantities of coarse and fine particles in a powder may be readily determined.

##### *Method A*

##### General Description

5. The test is made by distributing the powder on a series of sieves of different mesh openings and determining the weight of material retained by each sieve after thorough shaking.

##### Apparatus

6. (a) *Sieves*.—The sieves used shall be of the U. S. standard sieve series, half-height, 8 in. in diameter, conforming to the requirements of the Standard Specifications for Sieves for Testing

Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.<sup>3</sup> The following sieves together with a cover and bottom pan will be required:

U. S. Standard Sieve Series Number	Sieve Opening, Microns
No. 12.....	1680
No. 20.....	840
No. 40.....	420
No. 70.....	210
No. 140.....	105

(b) *Machine Sieving Device*.—There will also be required a mechanical sieve-shaking device and an automatic time switch or equivalent device.

(c) *Balance*.—A sensitive laboratory balance of 500-g. capacity, sensitive to 0.1 g., shall be used for weighing the sample and the residue retained on the sieves. Other accessories required are suitable brushes for cleaning the sieves, paper for collecting samples and any other items of equipment ordinarily used in making a sieve analysis.

##### Procedure

7. The sieves specified in Section 6 shall be stacked together in the order of size opening with the coarsest on top and the pan on the bottom. A 100-g. sample (Note 1) shall be accurately weighed and transferred to the top sieve of the stack which shall then be covered. The stack of sieves shall then be placed in the machine sieving device and shaken for 10 min.  $\pm$  15 sec. After shaking, the stack of sieves shall then be carefully separated, beginning at the top, and the amount of material retained on each sieve and in the bottom pan shall be determined by weighing. In transferring the powder from the sieve to the weighing scale, care shall be taken to brush clean from the back of the sieve all adhering particles. The cumulative percentage of original sample retained

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

on each sieve and in the bottom pan shall be calculated (Note 2).

### Report

8. The report shall include the following:

(1) the actual percentage of material retained on each sieve, and

(2) the cumulative total percentage of material retained on each sieve and in the bottom pan.

### *Method B*

### General Description

9. The test is made by distributing the powder on one coarse and one fine sieve and determining the weight of the material retained on each sieve and in the pan after thorough shaking.

### Apparatus

10. (a) *Sieves*.—The sieves used shall be of the U. S. standard sieve series conforming to the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.<sup>3</sup> The sieves required are the No. 12 (1680-micron) and No. 140 (105-micron) together with a cover and bottom pan.

(b) *Mechanical Sieving Device*.—There will also be required a mechanical sieve-shaking device and an automatic time switch or equivalent device.

(c) *Balance*.—A sensitive laboratory balance of 500-g. capacity, sensitive to 0.1 g., shall be used for weighing the sample and the residue retained on the sieves. Other accessories required are suitable brushes for cleaning the sieves, paper for collecting samples and any other items of equipment ordinarily used in making a sieve analysis.

### Procedure

11. The No. 12 sieve shall be stacked on the No. 140 sieve with the pan under-

neath. A 100-g. sample (Note 1) shall be accurately weighed and transferred to the No. 12 sieve, which shall then be covered. The stack of sieves shall then be placed in the machine sieving device and shaken for 10 min.  $\pm$  15 sec. After shaking, the sieves shall be carefully separated and the amount of material retained on each sieve and in the pan shall be determined by transferring to the scale and weighing separately the contents of each sieve and of the pan, avoiding losses as much as possible by brushing out all adhering material into the scale in each case. The weights shall be recorded and the percentages calculated for the following three fractions (Notes 2 and 3):

Percentage retained on No. 12 sieve,  
Percentage retained on No. 140 sieve, and  
Percentage in the pan.

### Report

12. The report shall include the two percentages of coarse and fine material, respectively, as calculated in Section 11. Note should be made of any unusually large particles found on the No. 12 sieve.

NOTE 1.—If necessary this test may be made on a sample of any size from 50 to 200 g. The weight of sample used shall be stated in the report.

NOTE 2.—Ordinarily there is a small loss of dust indicated by the cumulative total of actual weight being less than 100 per cent. If this loss is not over 2 per cent, the amount reported through the finest sieve shall be increased until the total of all portions of the sample equal 100 per cent. If the cumulative total of actual weight is less than 98 per cent, the weights and operations shall be carefully checked and the work repeated, if necessary.

NOTE 3.—The consumer will probably be interested in particle size only along lines rather definitely restricted as follows:

First, particles substantially larger than normal sieve size, such as might be present due to broken screen wire in the manufacturers' plant, would tend to stop up tablet machine hoppers, give irregular tablet weights, and rough or streaked molded surfaces. Ordinarily

a single coarse sieve provides a satisfactory check, that is, the No. 12 given in Method B.

Second, an excess of very fine particles would tend to cause excessive dusting, high bulk factor and irregular tablet weights due to failure to pour freely. (The amount may be determined by the use of the No. 140 sieve in Method B.) A certain amount of the finest particle sizes is usually desirable for improving finish and reducing bulk factor. Excessive fines may be indicated better by bulk factor and pourability than by sieve test.

### APPARENT DENSITY

#### Scope

13. The apparent density test is intended for the determination of the fluffiness or bulk of molding powder in

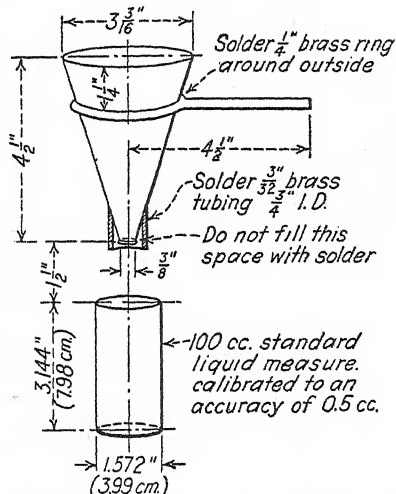


Fig. 1.—Apparatus for Apparent Density Test.

loose condition by weighing and measuring a standard quantity of the loose powder.

#### Apparatus

14. The apparatus shall consist of the following:

(a) *Measuring Cup*.—A cylindrical measuring cup of 100 cu. cm. capacity having a diameter equal to half the height. Such a cylinder would be 1.572 in. (3.99 cm.) in inside diameter by 3.144 in. (7.98 cm.) in inside height.

(b) *Funnel*.—A funnel mounted as shown in Fig. 1.

#### Procedure

15. A well-mixed 120-cu. cm. sample shall be selected from the general samples of powder. With the apparatus assembled as shown in Fig. 1, the small end of the funnel shall be closed with the hand or with a suitable flat strip of material and the 120-cu. cm. sample poured into the funnel. The bottom of the funnel shall then be opened quickly and the material allowed to flow freely into the cup. If caking occurs in the funnel the powder may be loosened with a small glass rod. After all the material has passed through the funnel, the excess powder on the top of the cup shall be scraped off with a straight edge. The material in the cup shall then be accurately weighed to the nearest 0.1 g. The weight, in grams, of 1 cu. cm. of powder shall then be calculated (Note 4). Three determinations of the apparent density shall be made on each sample and the results averaged (Note 5).

#### Report

16. The report shall include each value of apparent density and the average apparent density.

NOTE 4.—To convert grams per cubic centimeter to ounces per cubic inch multiply by 0.576. To convert grams per cubic centimeter to grams per cubic inch multiply by 16.38.

NOTE 5.—Apparent density figures are not comparable except on compounds having the same specific gravity after molding.

### BULK FACTOR

#### Bulk Factor

17. Bulk factor is the ratio of the volume of any given quantity of loose molding powder to the volume of the same material after molding. By suitable transformation of this ratio, it will be found that the bulk factor is also

equal to the ratio of the specific gravity after molding to the apparent density of the loose molding powder.

#### Procedure

18. (a) *Apparent Density*.—The apparent density of the molding powder shall be determined as described in Sections 13 to 16.

(b) *Specific Gravity*.—The specific gravity of the molding powder (after molding) shall be determined by any accurate method; such as, weighing two or more samples molded from the molding powder in air and in water. The average result obtained shall be used. If the shape of the piece is such that the volume may be readily calculated from the dimensions, it will be satisfactory to use this calculated volume in place of that determined by loss of weight in water.

(c) *Calculation*.—The bulk factor shall be calculated by dividing the average specific gravity of the molded piece by the average apparent density (in grams per cubic centimeter) of the molding powder.

#### Report

19. The report shall include the average apparent density and specific gravity and the bulk factor (Note 6) calculated from them.

NOTE 6.—The bulk factor test is comparable as between all grades of molding compound, regardless of the specific gravity after molding.

### POWDER POURABILITY

#### Scope

20. The powder pourability test is intended for determining how readily molding powders will feed through the hoppers and deliver uniform weights of material into the dies of tableting

machines by measuring the time of flow of a standard quantity through a standard funnel. The test is generally adaptable quantitatively only to materials of bulk factor 3.0 or less (see Section 17).

#### Apparatus

21. The apparatus shall consist of a funnel mounted as shown in Fig. 1, and a stop watch.

#### Procedure

22. A sample of the powder, equivalent to 100 times the molded specific gravity in grams (see Section 18 (b)) shall be taken (Note 7). This sample shall be worked on a paper until there is no tendency of the material to pack or cake. With the apparatus assembled, as shown in Fig. 1, the small end of the funnel shall be closed with the hand or with a suitable flat strip of material, and the sample poured lightly into the funnel, avoiding any tendency to pack it. The bottom of the funnel shall then be opened quickly, and the stop watch shall be started at the same instant. The powder shall be allowed to run from the funnel as freely as it will and the watch shall be stopped at the instant the last grains leave the funnel.

NOTE 7.—Correct mold charges are those weights which produce the constant molded volume of the piece to be molded and shall be so measured. Comparable quantities of material for this test are therefore some multiple of the molded specific gravity, 100 being taken as convenient.

#### Report

23. The report shall state the time in seconds required for the funnel to discharge; or, if so found, that the material will not run through the funnel (Note 8).

NOTE 8.—Molding powders which are not measurable by this test generally do not feed well without special feeders.

# Standard Method of Test for ACETONE EXTRACTION OF PHENOLIC MOLDED OR LAMINATED PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 494 - 46

ADOPTED, 1941; REVISED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 494; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method covers the procedure for determining the amount of acetone-soluble matter in molded or laminated phenolic products.

## Significance of Test

2. (a) For molded phenolic products, acetone extraction should be considered solely as a quantitative expression of a property normally associated with degree of cure. There is no demonstrably rigorous relation between the optimum mechanical and electrical properties of a well-cured piece and the numerical value of the acetone test. The amount of acetone-soluble matter is affected by: (1) Nature of resin and filler, (2) Lubricant, (3) Molding temperature, (4) Length of cure, (5) Thickness of the section from which sample is taken, (6) Nature of molded piece, (7) Technique used in molding, (8) Distribution of fines in the material to be extracted, and (9) Method of grinding specimen. These variations under some conditions may cause a difference of 3 to 4 per cent in

acetone extractable matter. For this reason, the method should be used only as a comparative test for measuring undercure.

(b) For laminated phenolic products, acetone extraction indicates change in stage of cure, change in resin content, change in type of resin used, presence of plasticizers or other acetone extractable addition agents, and is affected in general by the same factors as stated in Section 4 (a).

## Apparatus

3. (a) *Sieves*.—The set of sieves used shall consist of sieves Nos. 40 (420-micron) and 140 (105-micron), with a cover and receiving pan, conforming to the requirements of the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11) of the American Society for Testing Materials.<sup>3</sup>

(b) *Extraction Apparatus*.—The apparatus may be of the type shown in Fig. 1, or a Wiley-Richardson type, as shown in Fig. 2. The former type is more suitable for use with small electric hot plates while the latter is more suitable for use with oil or water baths. In either case,

<sup>1</sup> Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1938 to 1941, being revised in 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

it shall be possible to control the temperature so that the rate of extraction can be regulated accurately.

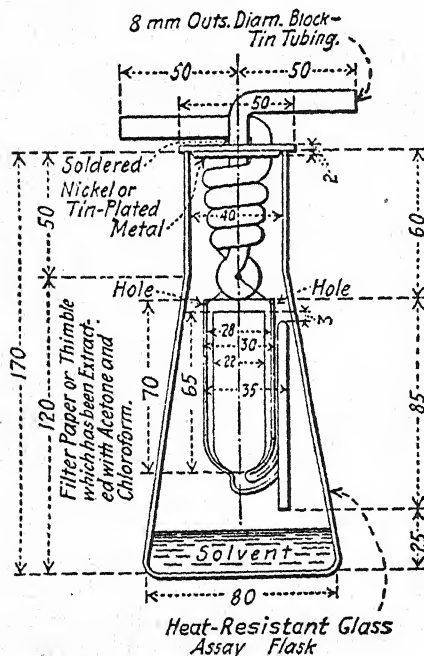
(c) *Drying Dishes*.—The drying dishes shall be lightweight dishes, approximately  $2\frac{1}{2}$  in. in diameter and  $1\frac{1}{2}$  in. in height.

#### Preparation of Sample

4. (a) *Precautions*.—It is of utmost importance that extreme care shall be

material shall occur which will tend to preclude the material.

(b) If it is impracticable to obtain samples by drilling, the parts may be broken up with a lathe, planer, milling machine, or a suitable grinder. A mortar and pestle or a pebble mill is considered suitable as a grinder, provided no perceptible heating occurs during the grinding procedure. A sharp file or rasp may be used for procuring the sample where the size or shape of the part is such that no other method is suitable.



(All dimensions in millimeters.)  
FIG. 1.—Extraction Apparatus.

taken during the preparation of the sample for extraction. The sample shall be drillings if possible, however, if not possible other suitable means of producing particles equivalent to drillings may be used. Drillings taken from a large molded product shall be truly representative of all sections of the part in proper proportions. The drills for sampling shall be kept sharp and so operated that no undue heating of the

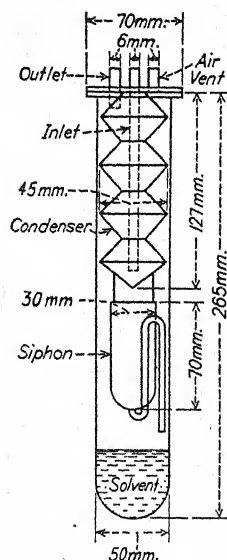


FIG. 2.—Wiley-Richardson Type Extraction Apparatus.

(c) In any case, the particles of the sample shall be of the smallest size practicable, so that they will pass through the No. 40 sieve with the minimum of reworking or grinding. It is important in preparing the sample that the smallest possible volume shall be obtained for a unit weight of the material.

(d) The sample shall be sieved through a No. 40 sieve and that part which will



not pass through shall be reground and blended with the original material passing the sieve. After assembling the Nos. 40 and 140 sieves and the receiving pan, the sample shall be placed in the top sieve, the cover placed on, and the entire sample shall be resieved either by a mechanical sieve shaker or hand sieving. If the hand-sieving method is used, the sieve shall be rotated with slight tapping, the period of rotation being 5 min.

(e) After sieving, the sample (that portion which has passed through the No. 40 sieve and has been retained on the No. 140 sieve) shall be placed immediately in an airtight container to prevent absorption of moisture by the powder and the consequent error in results.

#### Procedure

5. (a) *Extraction.*—The extraction procedure shall be carried out in triplicate. A 3,000-g. portion of the powdered sample shall be accurately weighed into a tared, open-texture quantitative filter paper,<sup>4</sup> 12.5 to 15 cm. in diameter, or a standard, single-thickness extraction thimble, 80 by 22 mm., trimmed if necessary. After folding over the thimble or filter paper containing the sample so that none of the powder can float out, it shall be placed in a desiccator until ready to insert in the siphon.

(b) The filter paper or thimble containing the weighed sample shall be pressed into the siphon in such a way that the outlet of the bottom is not plugged. The condenser and the siphon shall be placed in the extraction tube and 50 ml. of c.p. acetone added. The water shall be started through the condenser and the heat (Note 1) adjusted so that the siphon fills and empties between 15 and 20 times per hour. This rate shall be carefully maintained and the

sample shall be extracted for 4 hr. After the siphon empties, the flask shall be removed and the contents poured into an individually weighed dish. The flask shall be washed three times with the smallest possible quantity of acetone, using a wash bottle, and the washings shall be added to the extracted liquid in the dish.

NOTE 1.—If an oil or water bath is used for heating, the height of the liquid in the bath should not come above 1 in. below the highest level of the acetone in the siphon before the siphon starts to discharge.

(c) *Drying.*—The dish shall be placed in a well-ventilated drying chamber, maintained at  $50 \pm 2$  C. (Note 2), and the sample shall be dried to constant weight. Between dryings, all dishes containing the residue shall be kept in a desiccator to prevent the absorption of moisture.

NOTE 2.—It is very important that the specified temperature shall be maintained, otherwise consistent results can not be obtained between different laboratories. An electrically-heated oven should not be used unless it is exceedingly well ventilated as the acetone fumes are liable to come in contact with the heated coils and cause an explosion.

#### Calculation and Report

6. (a) *Calculation.*—The percentage of acetone extractable matter in the specimen shall be calculated as follows:

$$\text{Acetone extractable matter, per cent} = \frac{W - D}{S} \times 100$$

where:

$W$  = weight of the dish and extract,  
 $D$  = weight of the dish, and  
 $S$  = weight of the original sample.

(b) *Report.*—The report shall include the percentage of acetone extractable matter for each sample, and the average percentage of acetone extractable matter for the three samples.

<sup>4</sup> W. and R. Balston, Ltd., Genuine Whatman Filter Paper, No. 41H is suitable for this purpose.



# Standard Method of Test for PUNCHING QUALITY OF PHENOLIC LAMINATED SHEETS<sup>1</sup>



A.S.T.M. Designation: D 617 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 617; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. (a) This method of test covers the procedure for determining the punching quality of phenolic laminated sheets  $\frac{1}{8}$  in. and under in thickness. It involves the use of a standard punching die and standard punching practice to form punched specimens both at room temperature, designated as cold punching, and at an elevated temperature of 135 C., designated as hot punching, and a standard system for rating the punched specimens produced.

(b) This method further provides for a control test on uniformity of punching quality after this property has been determined with the punching die for a particular grade and thickness. The control test consists in the determination of Rockwell hardness at room temperature, and Rockwell hardness at 135 C. under a definitely prescribed procedure.

## Apparatus

2. The apparatus shall consist of the following:

(a) *Punching Die*.—A compound die

conforming to the dimensions prescribed in Figs. 1 and 2.

(b) *Oven*.—An oven which will provide a temperature of  $135 \pm 2$  C.

(c) *Hardness Tester*.—A Rockwell hardness tester conforming to the requirements prescribed in the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.<sup>3</sup>

## Test Specimens

3. (a) *Punching Test Specimens*.—For punching out specimens in the compound die, two strips of the material shall be used, each  $1\frac{1}{2}$  in. in width by 12 in. in length, one cut lengthwise and one cut crosswise from the sheet. The dimensions of the punched specimen are shown in Fig. 3.

(b) *Hardness Test Specimens*.—The specimen for the cold Rockwell hardness test and hot Rockwell hardness test shall conform to that prescribed in Section 37 of the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials.<sup>4</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1944.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Parts I-A and I-B.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Preparation of Apparatus

4. (a) The results obtained with this test method will depend upon the care taken in properly assembling the die (see Fig. 2) as well as in the condition of the punch and die. It is recommended that a standard die set be used in order to insure proper alignment of the punch and die at all times. The

The punch shall enter the die about  $\frac{1}{4}$  to  $\frac{1}{2}$  in. The clearances maintained between the punch and die shall be 0.002 in.

### Procedure for Punching

5. (a) Specimens shall be punched out individually by means of foot release of the press. Continuous punching of specimens shall not be used. The strips

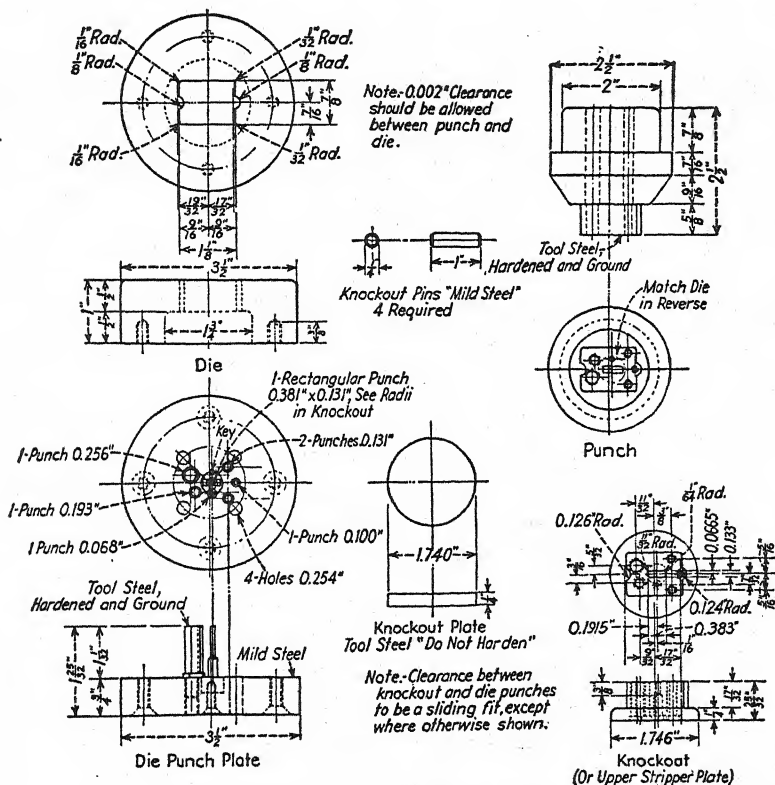


FIG. 1.—Compound Die for Punching Specimens.

cutting edges of the punches, as well as those of the die, shall be examined for sharpness. If the surfaces are not flat and if the edges are not sharp, it is advisable to grind the surfaces of both the punch and die to insure good punching.

(b) The punching die shall be set in the press in a vertical or slightly inclined position and shall be well aligned.

shall be so guided that the punched specimen is in the center of the strip. No specimens shall be punched closer together than twice the thickness of the strip. The length of stroke of the punch shall be between  $1\frac{1}{2}$  to 2 in., and the speed running idle shall be between 100 to 150 rpm.

(b) *Cold Punching.*—When strips are

punched at room temperature, the temperature shall be recorded. It is advisable to avoid punching the harder materials in thicknesses over  $\frac{3}{8}$  in. at room temperature, in order to prevent undue stress on the die.

(c) *Hot Punching*.—When strips are

The placing of these strips in the oven shall be so staggered that the heating time will be within the required period. (See Appendix I.)

### Rating Punching Quality

6. To evaluate punching quality, the

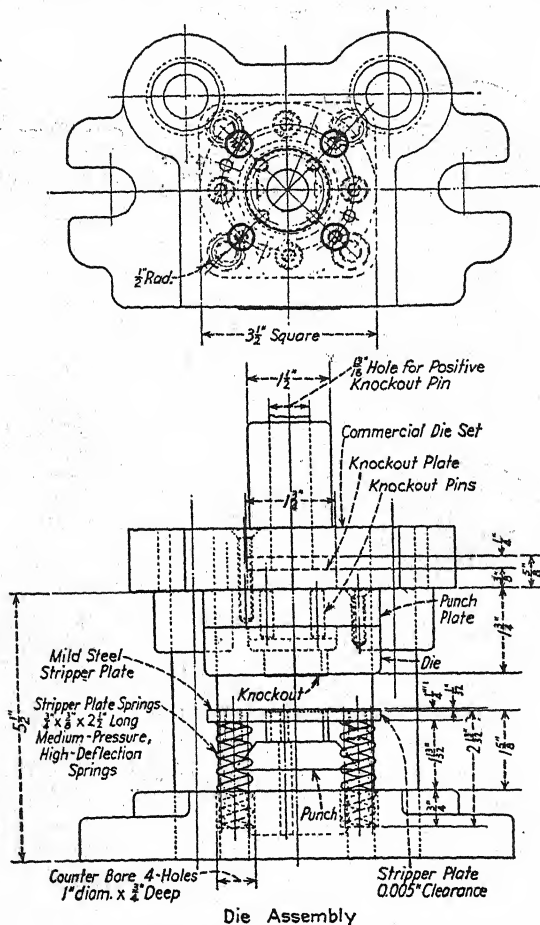


FIG. 2.—Die Assembly for Punching Quality Test.

punched at elevated temperatures, they shall be heated for 15 min. in an oven at  $135 \pm 2$  C., removed one at a time from the oven, and punched immediately. The strips shall be heated for not less than 15 min. nor more than 20 min.

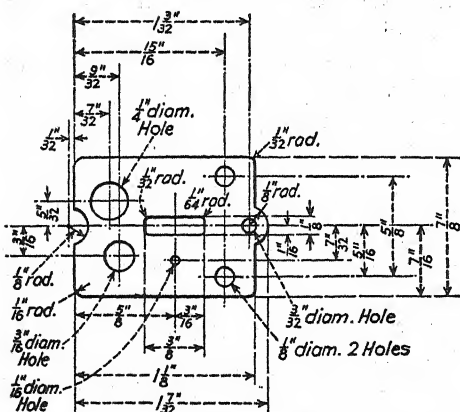
edges, surfaces, and holes of the punched specimens shall be rated separately in accordance with a point rating system ranging from 100 or excellent to 0 or worthless in accordance with Table I. The average of these three ratings on

each specimen shall constitute the measure of the punching quality of the material. (See Figs. 4, 5, and 6, in Appendix II illustrating typical characteristics of punched specimens and point ratings of these characteristics.)

punching quality. When the material is to be punched cold, the Rockwell hardness shall be determined at room temperature and when punched hot, the hardness shall be determined at an elevated temperature.

TABLE I.—POINT RATING OF PUNCHED SPECIMENS.

Rating	Point Score	Edge	Surface	Holes
Excellent	100....	Clean cut	Unaffected	Clean cut, no bulging
Very good	90....	Very slight chipping or very slight drag	Microscopic cracks around edges of piece or holes	Slight chipping in walls of holes or very slight bulging around hole
Good	80....	Slight chipping or slight drag	Very slight surface cracks around edge of piece or holes or slight raising of surface	Some chipping in walls of holes or slight bulging around hole
Fair	70....	Some chipping or drag	Slight surface cracks or some raising of surface around holes	Considerable chipping in walls of holes, some bulging around holes or slight tapering of hole
Poor	50....	Objectionable chipping or drag	Surface cracks around edges of piece or holes or distinct raising of surface	Bad chipping in walls of holes, pronounced bulging around hole, or pronounced tapering of hole
Very poor	25....	Bad chipping or slight cracking of edges	Pronounced surface cracks or pronounced raising of surface at edges or around holes	Slight cracks extending in body of material from holes, severe bulging around holes, or very pronounced tapering of hole
Worthless	0....	Severe chipping or cracking of edges	Very severe surface cracking or raising of surface	Body of material cracked at holes, very severe bulging at holes or closing up of hole with material



For Materials  $\frac{1}{8}$  to  $\frac{1}{4}$  in Thickness, Inclusive.  
Variations of  $\pm 0.005$  Permitted, Unless  
Otherwise Specified.

FIG. 3.—Punched Test Specimen.

### Procedure for Rockwell Hardness Control Test

7. (a) *Rockwell Hardness Test.*—Rockwell hardness may be used as a control test for keeping a check on the

(b) *Cold Rockwell Hardness.*—The cold Rockwell hardness shall be determined as described in Sections 36 to 39 of Methods D 229.<sup>4</sup>

(c) *Hot Rockwell Hardness.*—The hot Rockwell hardness shall be determined in general as described in Sections 36 to 39 of Methods D 229 with the following additional precautions necessitated by the elevated temperature:

(1) Enough pieces to make a  $\frac{1}{4}$ -in. pile-up for the test specimen shall be placed one layer deep in the oven at  $135 \pm 2$  C. for not less than 15 min. nor more than 20 min. Any pieces left in the oven for more than 20 min. shall be discarded, since any extra heating may cause an appreciable change in the properties of the material under test and lead to erroneous results.

(2) After heating for 15 min., the pieces shall be removed from the oven, piled up to form a specimen  $\frac{1}{4}$  in. in over-all thickness, and a stop watch shall be started the instant the pieces are

taken from the oven shelf. The specimen pile-up shall immediately be placed on the Rockwell anvil, and the minor load brought to bear by screwing up the anvil. At the instant the dial indicates zero, the major load shall be applied and as soon as fully applied, that is the instant the crank handle mechanism comes to a stop, the major load shall be removed. The number of seconds between the time the specimens were removed from the oven until the maximum hardness value is reached on the scale, shall be recorded together with the reading of the Rockwell hardness value to the nearest 0.5. In good practice, the time for the first test will be between 30 and 50 sec.

(3) The minor load shall then be released, and the specimen moved slightly so that depressions will be at least  $\frac{3}{16}$  in. apart. A second Rockwell hardness reading shall be taken and the time and the Rockwell hardness value to the nearest 0.5 at which an appreciable increase in scale reading ceases shall be recorded.

NOTE.—Do not wait too long for the needle to stop moving, since an error of 0.5 to 1.0 in the Rockwell hardness value will affect the curve less than 10 to 15 sec. error in time.

A third reading shall be taken followed by additional readings up to a period of 4 to 5 min. with a minimum of five readings. When five readings are taken, the third reading shall be completed between 110 and 130 sec., the fourth between 170 and 190 sec., and the last between 220 and 260 sec. A curve shall be plotted with

the time after removal from the oven as abscissas and the Rockwell hardness values as ordinates, using equal scale divisions for 10 Rockwell units and 50 sec. (See Appendix III for typical curve showing change of Rockwell hardness, M scale, with time after removal of the specimen from the oven.)

#### Report

8. (a) *Punching Quality Test.*—The report of the punching quality test shall include the following:

- (1) Thickness of specimen,
- (2) Point rating of each punched specimen for edge, surface, and holes, average rating for each of these three, and general average rating for punched specimens,
- (3) Room temperature for cold punching test, and
- (4) Oven temperature for hot punching test.

(b) *Control Test.*—The report on the Rockwell hardness test shall include the following:

- (1) Thickness of the specimen pile-up and number of layers of strip in the pile-up,
- (2) Cold Rockwell hardness, that is, the average of five readings for each grade of material tested,
- (3) Hot Rockwell hardness, 120 sec. after removal from oven as determined from the curve, that is, the average of two readings for each grade of material tested.
- (4) Room temperature for cold Rockwell test, and
- (5) Oven temperature for hot Rockwell test.

#### APPENDIX I

EXPLANATORY NOTE.—While the test temperature for the hot punching test of 135 C. for a 15-min. heating period serves to determine the relative hot punching qualities of various grades of phenolic laminated sheet, and is a satisfactory commercial punching condition for many grades, there are certain of the medium soft grades which may in some thicknesses give better commercial results when heated for shorter periods at 135 C., or when heated at a somewhat lower temperature.

APPENDIX II

ILLUSTRATIONS OF POINT RATINGS FOR DETERMINING PUNCHING QUALITY OF  
PHENOLIC LAMINATED SHEETS



$\frac{1}{16}$ -in. XP, Punched Cold  
95 points  
Edge Almost Clean-cut



$\frac{1}{16}$ -in. XXP, Punched Hot  
80 Points  
Slight Drag

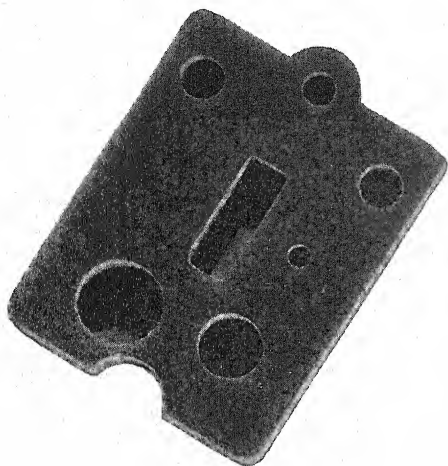


$\frac{1}{8}$ -in. XP, Punched Hot  
50 Points  
Objectionable Drag

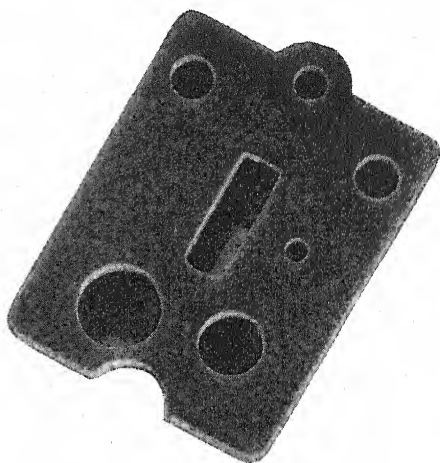


$\frac{1}{16}$ -in. XXX, Punched Cold  
25 Points  
Bad Chipping of Edges

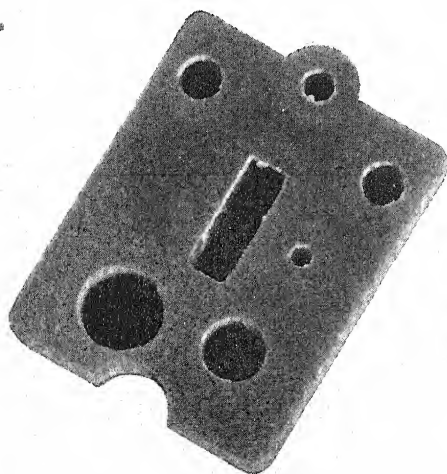
FIG. 4.—Illustrations of Point Ratings for Edges.



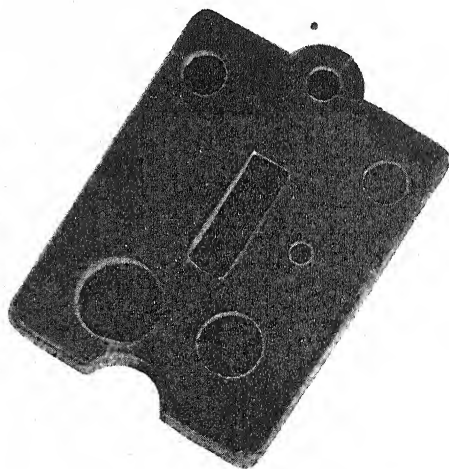
$\frac{1}{8}$ -in. XP, Punched Cold  
85 Points  
Extremely Slight Surface Cracks  
Around Holes



$\frac{1}{8}$ -in. XXP, Punched Hot  
75 points  
Very Slight Surface Cracks Around  
Holes and Edge of Piece



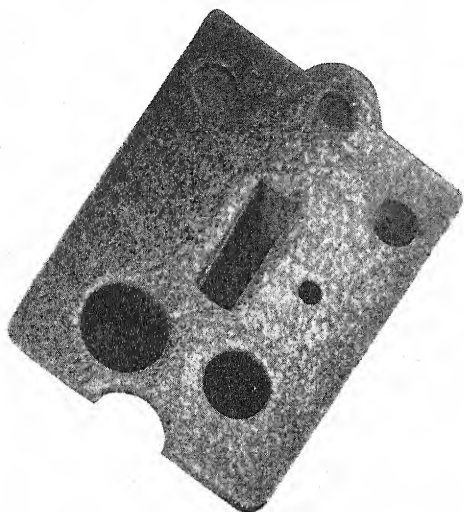
$\frac{1}{8}$ -in. XP, Punched Hot  
50 Points  
Distinct Raising of Surface Around  
Holes



$\frac{1}{8}$ -in. X-101, Punched Hot  
25 Points  
Pronounced Surface Cracks at Edges and  
Around Holes

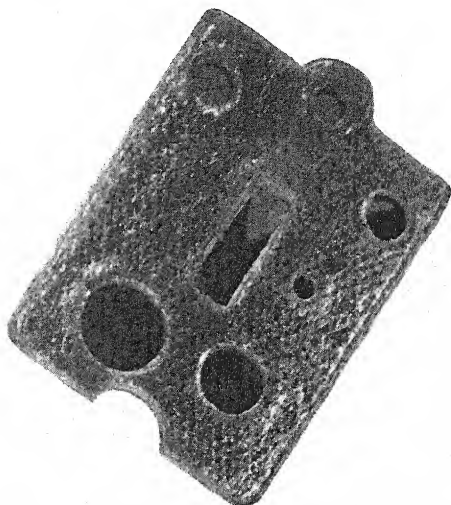
FIG. 5.—Illustrations of Point Ratings for Surfaces.





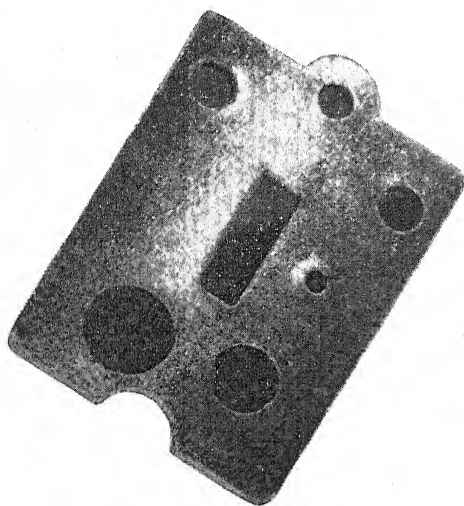
$\frac{1}{16}$ -in. XP, Punched Cold  
95 Points

Holes Clean-cut with Only Slight  
Bulging Around Smallest Hole



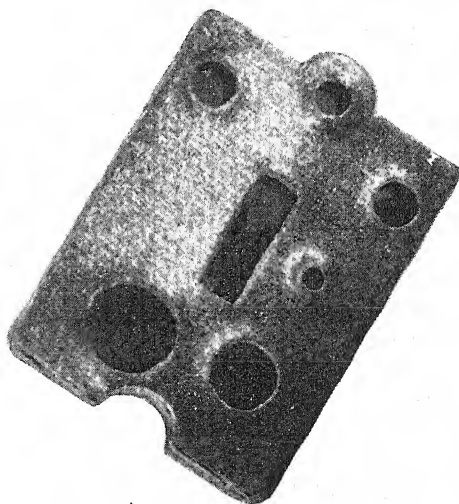
$\frac{1}{16}$ -in. XXP, Punched Hot  
80 Points

Holes Show Slight Drag with Some  
Bulging Around Holes



$\frac{1}{8}$ -in. XP-208, Punched Hot  
50 Points

Pronounced Bulging, Particularly  
Around Smaller Holes



$\frac{1}{8}$ -in. X-101, Punched Cold  
0 Points

Very Severe Bulging at Holes

FIG. 6.—Illustrations of Point Ratings for Holes.

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## APPENDIX III

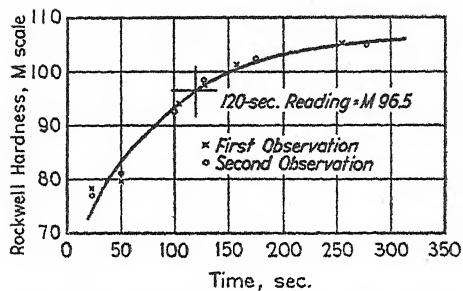


FIG. 7.—Typical Curve Showing Change in Rockwell Hardness, M Scale, with the Time After Removal of Specimen from Oven.

*Standard Method of*  
MEASURING SHRINKAGE FROM MOLD DIMENSIONS  
OF MOLDED MATERIALS USED FOR  
ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 551 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 551; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Purpose and Scope

1. (a) The purpose of this test method is to measure the batch-to-batch uniformity in initial shrinkage from mold to molded dimensions of either thermoplastic or thermosetting materials. The test specimen is a  $\frac{1}{2}$  by  $\frac{1}{2}$  by 5-in. bar, and positive or semipositive molds are used in which there is no very great movement of the plastic. Shrinkages measured by this procedure may not be representative of shrinkages to be expected in other molds or under other molding conditions, because shrinkages from mold to molded article may vary with how much or how little the charge is moved or flowed in the molds used, with molding conditions, with thickness of molded sections, etc. In general, flow or movement of the charge in the mold increases shrinkage. In general, decreasing the discharge temperature of the mold decreases shrinkage, and the

shrinkage of some plastics may be very sensitive to this temperature. Likeness in shrinkage by this procedure may not guarantee likeness in shrinkage in other molds or under other molding conditions, unless the samples or materials compared are alike in plasticity, since samples or materials of unlike plasticity may not have like shrinkages relative to each other in all molds and under all conditions. Primarily this test method is simply a test for batch-to-batch uniformity, and data from it should not be used to predict shrinkages elsewhere unless these other factors influencing shrinkage can be taken into consideration.

(b) This method does not provide for the measurement of shrinkages which may occur as molded materials age.

#### Sampling

2. Dependent upon the uniformity of the material to be tested, such samples as are necessary to represent it shall be taken and kept at room temperature in nominally airtight containers until

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

tested. If composite samples are desired, the method of sampling described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials<sup>3</sup> shall be followed.

#### Apparatus<sup>4</sup>

3. The apparatus shall consist of the following:

(a) *Molds*.—The single bar, single cavity positive mold shall conform to the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (A.S.T.M. Designation: D 647) of the American Society for Testing Materials.<sup>3</sup>

(b) *Press*.—A suitable hydraulic press.

(c) *Balance*.—A balance for weighing the mold charges.

(d) *Gages*.—Gages for measuring the molds and the test specimens.

#### Test Specimens

4. The test specimens shall be  $\frac{1}{2}$  by  $\frac{1}{2}$  by 5-in. bars as molded in accordance with Section 5.

#### Procedure

5. (a) The length of the mold cavity at room temperature shall be measured to the nearest 0.001 in. unless the length is already known.

(b) Sound test specimens shall be molded from the sample to be tested, using such conditions of pressure, temperature, time, etc., as the manufacturer and the purchaser may agree are suitable for the material. In the absence of other definite recommendations, the

following have been suggested as suitable molding procedures for various types of plastics:

*Thermoplastics*.—For thermoplastics, such as cellulose acetate, load the mold evenly at room temperature with the requisite quantity of granular material (also at room temperature), and place the mold in a hydraulic press such that a pressure of 3000 to 5000 psi. on the molded area can be applied and the mold can be heated to 250 to 265 F. (121 to 129 C.) in 5 to 10 min. Cool the mold to room temperature and discharge. The rate of heating, the maximum temperature reached, and the rate of cooling do not seem critical, but the temperature of discharge should be quite definite.

*Thermosetting Materials, Urea Type*.—From thermosetting materials of the urea type, load the mold evenly at 285 to 290 F. (141 to 143 C.) with the requisite quantity of granular material at room temperature, and place the mold in a hydraulic press such that the temperature can be maintained on the mold and a pressure of 3000 to 5000 psi. applied on the molded area. A release of pressure for 5 sec. after 5 to 15 sec. may be desirable. Discharge after 8 min. without cooling the mold.

*Thermosetting Materials, Phenolic Type*.—For thermosetting materials of the phenolic type, load the mold evenly at 300 to 310 F. (149 to 154 C.) with the requisite quantity of granular material at room temperature, and place the mold in a hydraulic press such that the temperature can be maintained on the mold and a pressure of 3000 to 5000 psi. applied on the molded area. Discharge after 6 min. without cooling the mold.

(c) After molding, the test specimens shall be allowed to cool to room temperature, and the length of each specimen shall be measured to the nearest

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> Editorially revised in November, 1945.

0.001 in. in not less than 16 nor more than 24 hr. after molding. A sufficient number of specimens shall be tested to give a reliable or reproducible average value for shrinkage.

#### Calculations and Report

6. (a) The shrinkage shall be calculated by subtracting the length of the specimen from the length of the mold cavity in which it was molded and dividing the difference by the latter.

(b) The report shall include the following:

(1) The molding procedure used,

including the form and distribution of the mold charge and the molding pressure, temperature, and time.

(2) The shrinkage from mold dimension expressed in inches per inch.

#### Reproducibility of Results

7. The probable limit of accuracy of this test is plus or minus 0.0005 in. per in. With this limitation in accuracy it is possible for samples to appear to be alike in shrinkage and actually to differ by 0.001 in. per in., or for samples to appear to differ by 0.001 in. per in. and actually to be alike.

# *Standard Methods of Test for*

## PRODUCT UNIFORMITY OF PHENOLIC LAMINATED SHEETS<sup>1</sup>



A.S.T.M. Designation: D 634 - 44

Adopted, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 634; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These methods cover the procedures for six tests which serve to check the uniformity of the properties of a particular type or grade of phenolic laminated material produced by a particular manufacturer, and to make evident variations in portions of one particular shipment, or in shipments made from time to time.

(b) These methods do not in themselves necessarily indicate the quality of a material, or in what degree it may be suited to a particular application. They indicate variations in the laminated product which have been caused by changes made purposely or inadvertently in the manufacturing process or in the raw materials used. In some cases they also serve as a guide to the quality of the product or its suitability for some particular application.

(c) All of the test procedures apply to fabric base laminated sheets, but the procedures for thread count and ply

thickness as described in Section 4, cannot generally be applied to paper base laminated sheets, and the procedure for dielectric strength as described in Section 7 will not in general be applicable to asbestos base materials which have poor natural electrical characteristics.

### Nature of Test

2. The following six methods of test are used for determining the variations in raw material or manufacturing process.

(a) *Thread Count and Ply Thickness*.—This test, applicable in general to fabric-base grades of material, indicates variations in count or weight of the fabric base used or radical changes in resin content of the material.

(b) *Acetone Extractable Matter*.—This test indicates change in stage of cure of the material, change in type of resin used, or presence of acetone extractable addition agents.

(c) *Water Absorption*.—This test indicates changes in resin content, type of resin or raw materials used, or degree of impregnation sufficient to cause changes in water absorption.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1941 to 1944.

(d) *Dielectric Strength Parallel with Laminations After Immersion in Water.*—This test indicates presence of conducting materials, overcure, or high moisture content in treated materials before pressing.

(e) *Hot Rockwell Hardness.*—This test indicates undercuring of material, or presence of plasticisers.

(f) *Density.*—This test indicates changes in type of resin used, in resin content, in the character of the sheet filler material used, or the addition of pigments, and similar material. It also indicates insufficient molding pressure, although presence of such a condition is rare in sheet materials.

### Sampling

3. A sheet shall be selected at random from a lot or shipment of material of a particular grade and thickness. A strip 3 in. in width shall be trimmed from two adjacent edges of the sheet and discarded; then the specimens required for the various tests shall be cut from the remainder in such a manner that no specimen shall be closer than 3 in. to the original edge of the sheet.

NOTE.—Since the purpose of the tests in these methods is to determine uniformity between sheets and shipments, it is important that variations in properties which sometimes occur at the edge or corner of a sheet shall be eliminated.

### Thread Count and Ply Thickness

4. (a) *Apparatus.*—A textile thread counter, or a 4 to 6-power microscope with a  $\frac{1}{2}$  or 1-in. square opening in the base shall be used.

(b) *Test Specimen.*—The test specimen shall consist of one piece sawed from the material 2 by 3 in. by the thickness. (This is the same specimen as is used for determining the dielectric strength (Section 7)).

(c) *Procedure for Thread Count.*—The number of threads per inch shall be

counted in each direction. It shall be noted whether either the warp or filling is composed of two or more plies twisted together or whether the threads are single or double laid, also whether or not the weave is plain, twill, or satin. This count shall be made on the surface and at the second or third ply from the surface, or at any ply that appears to have a different construction from the remainder of the material.

(d) *Procedure for Ply Thickness.*—All edges of the specimen shall be examined. At the section where visibility is best, the number of plies or layers in the total thickness shall be counted, and the total thickness of sheet measured with a micrometer. The total thickness of sheet shall be divided by number of plies to obtain the ply thickness.

(e) *Report.*—The report shall include the following:

(1) The average of three readings for thread count made in each direction of the material, reporting the highest thread count first.

Example.—Count  $\left\{ \begin{array}{l} 76 \text{ single,} \\ \text{double} \\ \text{laid} \\ 28, 2\text{ply} \end{array} \right\}$  plain weave.

If any of the plies differ in construction, report the thread count of each and give location and number of such plies.

(2) The average of the determinations of ply thickness to the nearest 0.0001 in.

(f) *Reproducibility of Results.*—Duplicate determinations by different operators on the same specimen should not differ by more than plus or minus 1 for counts under 40 threads per inch, and plus or minus 2 for counts of 40 threads per inch and over. Duplicate determinations of the number of plies by different operators on the same specimen should agree exactly, and determinations of ply thickness should agree within 0.0001 in. for specimens  $\frac{1}{8}$  in. and under in thickness and within

0.0004 in. for specimens over  $\frac{1}{8}$  in. in thickness.

### Acetone Extractable Matter

5. (a) *Procedure*.—The acetone extractable matter shall be determined in accordance with the Standard Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (A.S.T.M. Designation: D 494) of the American Society for Testing Materials.<sup>3</sup>

(b) *Reproducibility of Results*.—Duplicate determinations by different operators should not differ by more than plus or minus 0.5 per cent extractable matter for values under 5.0 per cent and plus or minus 1.0 per cent for values from 5.0 to 12.0 per cent.

### Water Absorption

6. (a) *Procedure*.—The water absorption shall be determined in accordance with the Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.<sup>3</sup> Comparison of the water absorption values for determining the degree of uniformity shall be made only between materials of the same nominal thickness.

(b) *Reproducibility of Results*.—Duplicate determinations by different operators should not differ by more than plus or minus 5 per cent of the observed value.

### Dielectric Strength Parallel with Laminations (Step-by-Step Method)

7. Except as prescribed below in Paragraphs (a) to (d), the dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>3</sup> Comparison of dielectric strength values for determining

degree of product uniformity shall be made only between materials of the same nominal thickness.

(a) *Apparatus*.—The electrodes shall consist of steel tapered pins, No. 3 standard tapered pin, 3 in. in length, and 0.156 in. in diameter at the small end, and 0.219 in. in diameter at the large end.

(b) *Test Specimens*.—The test specimens shall consist of pieces sawed from the material 2 by 3 in. by the thickness with the 3 in. direction coinciding with the machine or warp direction of the laminated sheet (Note). Two holes  $\frac{3}{16}$  in. in diameter and 1.0 in. between centers shall be drilled in each specimen and reamed to the standard taper. The center line of these holes shall coincide with the length of the specimen. Six specimens shall be provided, one for preliminary determination of short-time dielectric strength and the other five for the step-by-step test.

NOTE.—Machine direction is the lengthwise direction of the original paper used as base for the laminated sheet measured as it is taken from a roll. Warp direction is the direction of the warp threads in a fabric base sheet. It shall be the responsibility of the manufacturer to inform the purchaser as to which is the warp or machine direction of the sheets for any particular grade.

(c) *Conditioning*.—The specimens shall be immersed for a period of 48 hr. prior to testing in water maintained at a temperature of  $50 \pm 2$  C.

(d) *Procedure*.—The specimen shall be removed from the water, the surface wiped dry with a clean cloth, the electrodes inserted, and the dielectric strength of the specimen shall be determined under oil maintained at a temperature of  $50 \pm 2$  C.

(e) *Reproducibility of Results*.—Duplicate determinations by different operators should not differ by more than plus or minus 10 per cent of the observed value.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## Hot Rockwell Hardness

8. The hot Rockwell hardness shall be determined in general as described in Sections 36 to 39 of the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials<sup>3</sup> with the additional precautions prescribed below in Paragraphs (a) to (d) necessitated by the elevated temperature. Comparison of hot Rockwell hardness values for determining the degree of uniformity are in general valid for any thickness of materials using a  $\frac{1}{4}$ -in. pile-up specimen. For specimens over  $\frac{1}{4}$  in. in thickness, comparison shall be made only between materials of the same nominal thickness.

(a) *Apparatus*.—The Rockwell hardness tester shall be used, conforming to the requirements of the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials.<sup>4</sup> The  $\frac{1}{4}$ -in. ball and 100-kg. load, red scale as prescribed in Methods D 229 shall be used.

(b) *Test Specimen*.—The test specimen shall be 1 in. square with a thickness of at least  $\frac{1}{4}$  in. A pile-up of a minimum number of layers to a total thickness of at least  $\frac{1}{4}$  in. shall be used for thinner materials. For either the single specimen or pile-up, the edges shall be sanded or filed back so that there is no possibility of pieces being held up by a raised border. If the sheet is warped, specimens shall be sanded so that the surfaces in contact with the anvil or with each other are flat.

(c) *Heating*.—The specimens shall be placed one layer deep in a constant temperature oven at  $135 \pm 2$  C. for a period corresponding to the thickness of the specimen, as follows:

Thickness, in.	Heating Period, min.
$\frac{3}{16}$ and under	15
Over $\frac{3}{16}$ to $\frac{1}{4}$	20
Over $\frac{1}{4}$ to $\frac{3}{8}$	25
Over $\frac{3}{8}$	30

Any specimens left in the oven for more than the period prescribed for the material being tested shall be discarded, since any extra heating may cause an appreciable change in the properties of the material under test and lead to erroneous results.

(d) *Procedure*.—After the prescribed heating period, the specimen or number of specimens required for the  $\frac{1}{4}$ -in. pile-up shall be removed from the oven, and a stop watch shall be started the instant that the specimen is taken from the oven shelf. The specimen or pile-up shall immediately be placed on the Rockwell anvil and the minor load brought to bear by screwing up the anvil. At the instant the dial indicates zero, the major load shall be applied and as soon as fully applied, that is the instant the crank handle mechanism comes to a stop, the major load shall be removed. The number of seconds between the time the specimens were removed from the oven until the maximum value is reached on the scale shall be recorded together with the reading of the Rockwell hardness value to the nearest 0.5. In good practice, the time for the first test will be between 30 and 50 sec.

The minor load shall then be released, and the specimen moved slightly so that depressions will be at least  $\frac{3}{16}$  in. apart. A second Rockwell hardness reading shall be taken and the time and the Rockwell hardness value to the nearest 0.5 at which an appreciable increase in scale reading ceases shall be recorded.

NOTE.—Do not wait too long for the needle to stop moving, since an error of 0.5 to 1.0 in the Rockwell hardness value will affect the curve less than 10 to 15 sec. error in time.

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Parts I-A and I-B.



A third reading shall be taken followed by additional readings up to a period of 4 to 5 min., with a minimum of five readings. When five readings are taken, the third reading shall be completed between 110 and 130 sec., the fourth between 170 and 190 sec., and the last between 220 and 260 sec. A curve shall be plotted with the time after removal from the oven as abscissas and the Rockwell hardness values as ordinates, using equal scale divisions for 10 Rockwell units and 50 sec.

(e) *Report.*—The report shall include the following:

- (1) The Rockwell hardness values read from the curve for the first 60-sec. and 120-sec. periods,
- (2) The temperature of the oven and the room,
- (3) The thickness of the sheet, and
- (4) The number of layers in the pile-up.

(f) *Reproducibility of Results.*—Duplicate determinations by different operators should not differ by more than plus or

minus 5 hardness values on the Rockwell scale.

### Density

9. (a) *Test Specimens.*—Test specimens shall consist of three pieces of the material each 3 by 1 in. by the thickness. The test specimens for the water absorption test (see Section 6) may be used for this test prior to making the water absorption test.

(b) *Procedure.*—Three specimens shall be tested. Any suitable weight difference apparatus for making readings in water and in air may be used in determining the density. The water used shall be maintained at  $25 \pm 2$  C., and correction shall be made for the density of the water.

(c) *Report.*—The density of the specimen at 25 C. in grams per cubic centimeter shall be reported.

(d) *Reproducibility of Results.*—Duplicate determinations by different operators should not differ by more than plus or minus 0.005 g. per cu. cm.

# Standard Methods of TESTING LAMINATED ROUND RODS USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 349 - 46

ADOPTED, 1939; REVISED, 1942, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 349; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods cover the procedures for testing laminated round rod materials to be used as electrical insulation.

## Conditioning

2. In order to eliminate the effects of previous history of humidity exposure and to obtain reproducible results (Note 1), the test specimens of laminated rods shall in all cases of dispute be given a conditioning treatment for physical tests (Note 2) as follows:

*Tensile, Flexural, and Compressive Strengths, and Density.*—Prior to test the machined specimens shall be conditioned by drying in an air-circulated oven at  $50 \pm 3$  C. for 48 hr., followed by cooling to room temperature in a desiccator. All specimens shall be tested at room temperature maintained at  $25 \pm 8$  C.

NOTE 1.—Conditioning of specimens may be undertaken: (a) for the purpose of bringing the material into equilibrium with normal or average room conditions of 25 C. and 40 per cent relative humidity; (b) simply to obtain reproducible results, irrespective of previous history of exposure; or (c) to subject the material to abnormal conditions of temperature or humidity in order to predict its service behavior.

The conditions given here to obtain reproducible results may give physical values somewhat higher or somewhat lower than values under equilibrium at normal conditions, depending upon the particular material and test. To insure substantial equilibrium under normal conditions of humidity and temperature, however, will require from 20 to 100 days or more depending upon thickness and type of material and its previous history. Consequently, conditioning for reproducibility must of necessity be used for general purchase specifications and product control tests.

NOTE 2.—Conditioning of specimens for electrical tests is also necessary to obtain consistent results. The procedure for such conditioning is under consideration by the committee. In order to secure comparative results, specimens should be conditioned at the same temperature and humidity.

## TENSILE STRENGTH

### Apparatus

3. Any universal testing machine may be used provided it is accurate to

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1932 to 1939, being revised in 1934, 1935, and 1939.

1 per cent of the lowest load to be applied. Jaws which tighten under load, such as wedge grip jaws, shall be used with the specimen properly aligned.

#### Test Specimens

4. The test specimens shall be about 12 in. in length. When the diameter of the rod is  $\frac{1}{4}$  in. or over, a groove 1 in. in width and  $\frac{1}{8}$  in. in depth with corners rounded to a radius of 3 in. shall be made around the rod at the center.

#### Procedure

5. (a) Five specimens shall be tested.

(b) *Speed of Testing*.—The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. when the machine is running idle.

#### Report

6. The report shall include the following:

(1) The average diameter of the specimen expressed to the nearest 0.001 in. determined from at least two measurements 90 deg. apart,

(2) The breaking load of each specimen in pounds,

(3) The tensile strength of each specimen in pounds per square inch, and

(4) The room temperature.

#### FLEXURAL STRENGTH

##### Apparatus

7. Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied.

##### Test Specimens

8. The test specimen shall have a diameter equal to the diameter of the rod and a length of 5 in.

#### Procedure

9. (a) Five specimens shall be tested. Each specimen shall be tested as a simple beam loaded at the center. The supports shall have contact edges rounded to a radius of  $\frac{1}{8}$  in. The distance between the points of support shall be 4 in. when the diameter of rod is  $\frac{1}{2}$  in. or over. For smaller rods the distance between supports shall be eight times the diameter of the rod. The load shall be applied through a steel block having a semicircular contact edge of the same radius as the rod with edges rounded to a radius of  $\frac{1}{8}$  in.

(b) *Speed of Testing*.—The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in per min. when the machine is running idle.

#### Report

10. The report shall include the following:

(1) The diameter of the specimen expressed to the nearest 0.001 in., determined from at least two measurements 90 deg. apart,

(2) The breaking load of each specimen in pounds, and

(3) The maximum fiber stress in pounds per square inch, calculated according to the following formula:

$$S = \frac{8 WL}{\pi d^3}$$

where:

$S$  = maximum fiber stress,

$W$  = breaking load in pounds,

$L$  = distance between supports in inches, and

$d$  = diameter in inches.

#### COMPRESSIVE STRENGTH

##### Apparatus

11. Any universal testing machine may be used provided it is accurate to

1 per cent of the lowest load to be applied. One end of the specimen shall bear upon an accurately centered spherical bearing block, located, whenever practicable, at the top. The metal bearing plates shall be directly in contact with the ends of the test specimen.

### Test Specimens

12. (a) Samples shall be tested as received. For rods  $\frac{1}{8}$  to  $1\frac{1}{2}$ -in. in diameter the test specimen shall have a diameter equal to the diameter of the rod and length conforming to the following requirements:

Diameter, in.	Length, in.
$\frac{1}{8}$ to $\frac{1}{2}$ .....	$\frac{1}{2}$
Over $\frac{1}{2}$ to $1\frac{1}{2}$ .....	1

(b) For rods over  $1\frac{1}{2}$  in. in diameter, specimens shall be 1-in. cubes cut from the rods so as to be representative of its cross-section both at the center and near the edges.

(c) The ends of each specimen shall be accurately cut or ground square with the sides.

### Procedure

13. (a) Five specimens shall be tested with the load applied perpendicular to the faces or ends of the specimen.

(b) *Speed of Testing.*—The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in per min. when the machine is running idle.

### Report

14. The report shall include the following:

(1) The diameter of the specimen expressed to the nearest 0.001 in., determined from at least two measurements 90 deg. apart,

(2) When 1-in. cube specimens are used, the dimensions and approximate location of each with reference to area of rod section,

(3) The load on each specimen in pounds at the first sign of rupture, and

(4) The ultimate compressive strength in pounds per square inch calculated from the data obtained on the application of the load perpendicular to the face of the specimen.

### WATER ABSORPTION

#### Procedure

15. The rate of water absorption shall be determined in accordance with the Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.<sup>3</sup>

### DENSITY

#### Test Specimens

16. Any suitable size specimen may be used. The samples used for the compressive strength test (Section 12) will be found convenient.

#### Procedure

17. Two specimens shall be tested. Any suitable weight-difference apparatus for making readings in water and in air, or the weight and dimensions of the specimen may be used as a basis for calculating the density.

#### Report

18. The report shall express the density in c.g.s. units.

### DIELECTRIC STRENGTH

#### Dielectric Strength

19. (a) Except as specified below in Sections 20 to 25, the dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149).<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) Tests shall be made parallel with the major axis of the rod.

### Electrodes and Test Specimens

20. The test specimens shall be  $\frac{1}{2}$  in. in length. A hole shall be drilled into one end of the test specimen in the approximate center of the rod parallel with the major axis of the rod to a depth of  $\frac{7}{16}$  in. leaving a section  $\frac{1}{16}$  in. in thickness to be tested. A snug-fitting metal pin electrode with the end ground to conform with the shape of the drill used shall be inserted in the hole. The specimen shall be placed on a flat metal plate having a diameter at least  $\frac{1}{2}$  in. greater than that of the specimen. This plate shall serve as the lower electrode. Thus, in effect, the material shall be tested parallel with laminations in a point-plane gap. The diameter of the hole shall be as shown in the following table:

Nominal Diameter of Rod, in.	Nominal Hole Diameter for Pin Electrode, in.
$\frac{1}{8}$ to $\frac{1}{4}$ , incl.	$\frac{1}{16}$
Over $\frac{1}{4}$	$\frac{1}{8}$

NOTE.—For rods with exceptionally high dielectric strength, surface flashover may occur. Such cases shall be noted in the report.

### Conditioning

21. (a) Unless otherwise specified, all test specimens shall be conditioned for 48 hr. at  $50 \pm 3$  C. in a circulating air oven prior to testing. After removal from oven, specimens shall be permitted to cool to room temperature in a desiccator over anhydrous calcium chloride.

(b) In the case of rods to be used at other than room temperature, the dielectric strength characteristics shall be determined over the operating range of temperature. Prior to test, specimens previously conditioned as described in Paragraph (a) shall be exposed to each test temperature in a suitable temperature-control chamber for a period in minutes equal to one half the diameter of the specimen in mils.

### Surrounding Medium

22. The specimens shall be tested immersed in oil, maintained at the temperature of the test specimen.

### Procedure

23. (a) Tests shall be made by either the short-time method or the step-by-step method.

(b) In tests made by the short-time method, the voltage shall be increased at the rate of 0.5 kv. per sec.

In tests made by the step-by-step method, the voltage at each step shall be applied for 1 min. The voltage shall be increased in increments as follows:

Breakdown Voltage by Short-Time Method	Increment of Increase of Test Voltage
25 kv. or less	1.0 kv.
Over 25 to 50 kv., incl.	2.0 kv.
Over 50 to 100 kv., incl.	5.0 kv.
Over 100 kv.	10.0 kv.

### Number of Tests

24. At least five tests shall be made at each temperature in the short-time method, and at least three tests in the step-by-step method. When the range of test temperatures is considerable, tests should be made at not less than five temperatures, if a curve of dielectric strength against temperature is desired.

### Report

25. The report shall include the following:

(1) A description of the material including name, type, grade, color, size, and name of the manufacturer.

(2) The conditioning treatment which the specimens have received.

(3) A statement of the procedure used whether short-time method, or step-by-step method.

(4) The maximum, minimum, and average puncture voltage in kilovolts and volts per mil (Note).

(5) Duration of test if step-by-step method, including the initially applied voltage in kilovolts.

(6) The temperature of the test specimen.

(7) The size and type of electrodes.

NOTE.—The thickness of the section shall be measured prior to breakdown. This can be conveniently done by measuring the length of the electrode, then the combined length of the specimen and electrode with the electrode inserted in place.

# Standard Methods of TESTING SHEET AND PLATE MATERIALS USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 229 - 46

ADOPTED, 1939; REVISED, 1942, 1943, 1946<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 229; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods cover the procedures for testing stiff, flat sheet and plate materials, such as phenolic and other types of laminated sheets, hard rubber, asbestos composition board, etc., to be used as electrical insulation except as modified by the individual methods or specifications for a particular material.

NOTE.—For tests applying to vulcanized fibre reference should be made to the Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation (A.S.T.M. Designation: D 619) of the American Society for Testing Materials.<sup>3</sup>

## Definitions

2. In referring to the cutting of the specimens and the application of the load, the following definitions apply:

*Flatwise.*—Load applied to the flat side of the original sheet or plate.

*Edgewise.*—Load applied to the edge of the original sheet or plate.

*Lengthwise.*—In the direction of the length of the sheet.

*Crosswise.*—In the direction at right angles to the length of the sheet.

NOTE.—When the sheet has the same length and width, one dimension shall arbitrarily be designated as the length, and the other as the width.

## Conditioning

\*3. The test specimens (Note 1) of flat sheet and plate materials shall be given a conditioning treatment for physical tests (Note 2) in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials,<sup>3</sup> modified as follows:

(a) The following materials shall be considered as being slowly affected by humidity and, unless otherwise noted, shall be conditioned in accordance with Functional Procedure A:

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1925 to 1939, being revised in 1926, 1928, 1930, 1931, 1932, 1934, 1935, 1936, 1937, and 1938.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(1) Laminated sheets bonded with phenolic, urea, melamine, or other thermosetting resins,

(2) Methyl methacrylate sheets,

(3) Asbestos boards, and

(4) Hard rubber.

(b) The following shall be considered as rapidly affected by relative humidity and, unless otherwise noted, shall be conditioned in accordance with the Standard Procedure:

(1) Pressboard and other cellulosic board, and

(2) Sheets of cellulosic esters and ethers.

(c) Sheet and plate materials which are covered by A.S.T.M. specifications shall be conditioned and tested by the procedures recommended in those specifications. Other sheet materials not covered by A.S.T.M. specifications or by Paragraphs (a) and (b) shall be conditioned according to the Standard Procedure and tested under the Standard Laboratory Atmosphere.

(d) *Impact Strength*.—The machined specimens of sheet materials shall be conditioned before testing in accordance with the Standard Procedure or Functional Procedure A, except that under Functional Procedure A materials may be tested, after cooling in a desiccator, as soon as room temperature has been reached. Following the conditioning as in Functional Procedure A, tests shall be made at room temperature maintained at  $25 \pm 8$  C., except where closer limits are specified.

(e) *Tensile, Flexural, Compressive, and Bonding Strengths, and Rockwell Hardness*.—For the materials listed in Paragraph (a) no conditioning is necessary except in case of dispute, in which case Functional Procedure A shall be followed, modified as prescribed in Paragraph (d). For materials requiring conditioning under the Standard Procedure, the exact

details prescribed in A.S.T.M. Methods D 618 shall be followed.

NOTE 1.—Conditioning of specimens may be undertaken: (1) for the purpose of bringing the material into equilibrium with normal or average room conditions of 25 C. (77 F.) and 40 per cent relative humidity, (2) simply to obtain reproducible results, regardless of previous history of exposure, or (3) to subject the material to abnormal conditions of temperature or humidity in order to predict its service behavior.

The conditioning prescribed in these methods to obtain reproducible results may give physical values somewhat higher or somewhat lower than values under equilibrium at normal conditions, depending upon the particular material and test. To insure substantial equilibrium under normal conditions of humidity and temperature, however, will require from 20 to 100 days or more depending upon thickness and type of material and its previous history. Consequently, conditioning for reproducibility must of necessity be used for general purchase specifications and product control tests.

NOTE 2.—Conditioning of specimens for electrical tests is also necessary to obtain consistent results. The procedure for such conditioning is under consideration by the committee. In order to secure comparative results, specimens should be conditioned at the same temperature and humidity.

## THICKNESS

### Apparatus and Procedure

4. (a) *On Test Specimens*.—In the test methods which follow, all thickness measurements shall be made using a machinist's micrometer in the manner outlined under method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

(b) *On Large Sheets*.—In the factory,<sup>4</sup> measurements of large sheets of material may be made by using a gage mounted on a yoke of sufficient size and rigidity to permit accurate measurements in the center of the sheet.

<sup>3</sup> Results of comparative tests in several factories, measuring 36-in. square sheets by a variety of such devices, indicate that the trade is able to measure sheets  $\frac{1}{8}$  and  $\frac{1}{4}$  in. in thickness to an accuracy of 0.0015 in. (In the tests,  $\sigma$ , or root mean square deviations, of 0.0005 in. were obtained.)



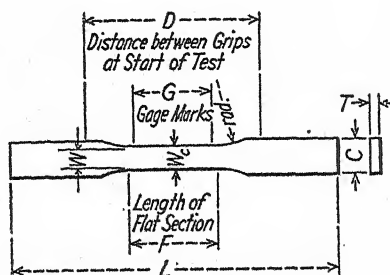
## TENSILE STRENGTH

## Tension Tests

5. Except for conditioning as specified in Section 3 and for the description of test specimens as specified in Section 6, the tensile properties, including both ultimate strength and modulus of elasticity, shall be determined in accordance with the Tentative Method of Test for

width at the center  $W_c$  shall be plus 0.000 in., minus 0.004 in. compared with the width  $W$  at other parts of the reduced section. Any reduction in  $W$  at the center shall be gradual with no abrupt change and equal on each side.

(b) Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested.



Dimension, in.	Thickness, <sup>a</sup> $T$ , in.					Tolerances, in.
	$\frac{1}{4}$ in. or under		Over $\frac{1}{4}$ to $\frac{1}{2}$ in., Incl.		Over $\frac{1}{2}$ to 1 in., Incl. <sup>b</sup>	
	Type I	Type II <sup>c</sup>	Type I	Type II <sup>c</sup>	Type I	
C—Width overall.....	0.750	0.750	1.125	1.125	1.500	$\pm 0.016$
W—Width of flat section.....	0.500	0.250	0.750	0.375	1.000	$-0.000$ $+0.005$
F—Length of flat section.....	2.25	2.25	2.25	2.25	2.25	$\pm 0.016$
G—Gage length <sup>d</sup> .....	2.00	2.00	2.00	2.00	2.00	$\pm 0.016$
D—Distance between grips <sup>d</sup> .....	$4\frac{1}{2}$	$5\frac{1}{2}$	$4\frac{1}{2}$	$5\frac{1}{2}$	$5\frac{1}{2}$	$\pm \frac{1}{8}$
L—Length overall.....	$8\frac{1}{2}$	$9\frac{1}{2}$	$9\frac{1}{2}$	$10\frac{1}{2}$	12	minimum
Rad.—Radius of fillet.....	3	3	3	3	3	minimum

<sup>a</sup> Tolerance on thickness shall be the standard for the grade of material tested.

<sup>b</sup> For sheets of a nominal thickness over 1 in. the specimens shall be machined to  $1.000 \pm 0.010$  in. in thickness. For thicknesses between 1 and 2 in. approximately equal amounts shall be machined from each surface. For thicker sheets both surfaces shall be machined and the location of the specimen with reference to the original thickness shall be noted.

<sup>c</sup> The type II specimen shall be used for material from which the type I specimen does not give satisfactory failures in the gage length, such as for resin-impregnated compressed laminated wood.

<sup>d</sup> Test marks only.

FIG. 1.—Tension Test Specimen for Sheet and Plate Insulating Materials.

Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

## Test Specimens

6. (a) Test specimens shall conform to the dimensions shown in Fig. 1. No transverse scratches or grooves shall exist in the reduced section. Care shall be taken in machining to see that the 3-in. radius sections are not undercut. The

## FLEXURAL STRENGTH

For Materials  $\frac{1}{32}$  in. or Over in Thickness<sup>5</sup>

## Apparatus

7. (a) Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied.

<sup>5</sup> Conventional flexure tests in a flatwise direction are not recommended for materials thinner than  $\frac{1}{16}$  in. nor in the edgewise direction for materials thinner than  $\frac{1}{4}$  in.

(b) The specimen shall be tested as a simple beam loaded at the center. The supports and loading nose shall have contact edges rounded to a radius of  $\frac{1}{8}$  in. for material  $\frac{1}{8}$  in. or over in thickness, and to a radius of  $\frac{1}{16}$  in. for thinner materials. For material thinner than  $\frac{1}{8}$  in. both supports shall be provided with a pin or other stop to align one side of the specimen perpendicular to the line of contact with the supports. The distance between the points of support shall be 4 in. for edgewise tests of all thicknesses and eight times the nominal thickness of the material for tests in the flatwise direction, except that the minimum span shall be  $\frac{1}{2}$  in.

#### Test Specimens

8. The test specimen shall be  $\frac{1}{2}$  in. in width except for specimens over  $\frac{1}{2}$  in. in thickness tested in the flatwise direction, which shall have the width equal to the thickness of the specimen. The thickness shall be the full thickness of the sheet. The length shall be 1 in. greater than the distance between the points of support as specified in Section 7 (b).

#### Procedure

9. (a) The specimens shall be tested as follows:

(1) Five specimens cut lengthwise of the sheet, tested flatwise,

(2) Five specimens cut lengthwise of the sheet, tested edgewise,

(3) Five specimens cut crosswise of the sheet, tested flatwise, and

(4) Five specimens cut crosswise of the sheet, tested edgewise.

(b) *Speed of Testing*.—The crosshead speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. per min. when the machine is running idle.

#### Report

10. The report shall include the following:

(1) The directions of cutting and loading of the specimen as specified in Section 9,

(2) The thickness and width of each specimen in inches expressed to the nearest 0.001 in. or in centimeters expressed to the nearest 0.025 mm.,

(3) The room temperature,

(4) The breaking load of each specimen in pounds or kilograms, and

(5) The maximum fiber stress in pounds per square inch or kilograms per square centimeter calculated as follows:

$$S = \frac{3 Pl}{2 bd^2}$$

where:

$S$  = maximum fiber stress,

$P$  = breaking load in pounds or kilograms,

$l$  = distance between supports in inches or centimeters,

$b$  = width of specimen in inches or centimeters, and

$d$  = depth of specimen in inches or centimeters.

#### COMPRESSIVE STRENGTH

##### Apparatus

11. The apparatus shall consist of the following:

(a) *Testing Machine*.—Any universal testing machine of the constant rate of crosshead movement type may be used provided it is accurate to 1 per cent of the lowest load to be applied.

(b) *Compression Tool*.—A compression tool for applying the load to the test specimen. This tool shall be so constructed that loading is truly axial, and so that the load is applied through surfaces which are flat and parallel to the ends (or faces) of the specimen and are normal to the vertical axis of the speci-

men. A suitable form of compression tool is shown in Fig. 2 and consists of the following:

(1) *Base Plate*.—A hardened steel base plate, the upper surface of which is machined and polished to as flat a surface as nearly perfect as possible.

(2) *Plunger and Ball*.—A hardened steel plunger, the lower end of which is machined and polished to a flatness as nearly perfect as possible and the upper end of which is machined and polished to a hollow hemisphere to receive a

framework. It shall be smooth and provided with adequate lubrication so that negligible force is required to move the plunger freely through the bushing.

(5) In use, the base of this compression tool shall be centered on the lower platen of the testing machine and contact with the top platen (or crosshead) shall be made between the hardened steel ball and a hardened steel plate attached to the top platen.

### Test Specimens

12. (a) For sheets 1 in. in thickness or over, the test specimen shall be a 1-in. cube, the faces of which shall be flat and perpendicular to the axes, being ground to this condition if necessary.

(b) For sheets less than 1 in. in thickness, the specimen shall consist of a pile of sheets 1 in. square with a minimum number of layers to produce a height of at least 1 in.

### Procedure

13. (a) For sheets 1 in. in thickness or over, three specimens shall be tested in the condition in which they are received with the load applied flatwise, three specimens with the load applied edge-wise-lengthwise of the sheet, and three specimens with the load applied edge-wise-crosswise of the sheet.

(b) For sheets less than 1 in. in thickness, three specimens shall be tested in the condition in which they are received with the load applied flatwise of the sheet.

(c) *Speed of Testing*.—The load shall be applied on the ends of the specimen and the crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. when the machine is running idle. For the best results the slowest possible speed should be used.

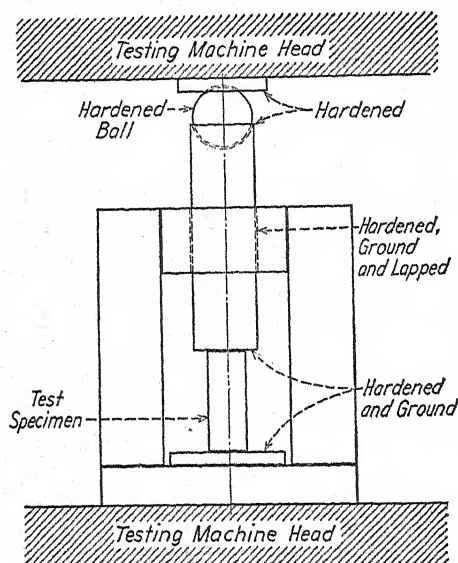


FIG. 2.—Compression Tool.

polished, hardened steel ball, the diameter of which approaches that of the plunger.

(3) *Guiding Framework*.—A guiding framework attached to the base plate and holding the plunger so that its longitudinal axis is perpendicular to the surface of the base plate and so that its polished lower surface is parallel to the polished surface of the base plate within 0.001 in. over the entire contact area.

(4) *Bushing*.—A hardened steel bushing holding the plunger in the guiding

## Report

14. The report shall include the following:

(1) The dimensions of each specimen in inches expressed to the nearest 0.001 in. or in centimeters expressed to the nearest 0.025 mm.,

(2) The direction of application of the load,

(3) The load on each specimen in pounds or kilograms at the first sign of failure,

(4) The ultimate compressive strength in pounds per square inch or kilograms per square millimeter, of each specimen, calculated from the area of each specimen measured before the load is applied,

(5) The general character of the material tested, with a description of how it acts under the applied load, and

(6) The room temperature.

## RESISTANCE TO IMPACT

### Test Specimens

15. Specimens for the Charpy impact test may be notched or unnotched, as desired, according to conditions of service, and for the Izod impact test shall be notched (Note). Specimens shall be cut both lengthwise and crosswise of the sheet.

NOTE.—The unnotched specimen gives a higher strength than the notched specimen, and the ratio of the strength of the unnotched specimen to that of the notched specimen varies with different materials.

### Procedure

16. Resistance to impact shall be determined in accordance with the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup>

## WATER ABSORPTION

### Purpose

17. The water absorption tests are intended to determine the rate at which

water is absorbed by the material when immersed and the total quantity absorbed at saturation.

NOTE.—The electrical properties of different materials are not necessarily affected to the same extent by the same increase in moisture content so that the water absorption test must be correlated with the desired electrical tests.

The water immersion test and exposure to air of high humidity are not always directly comparable and should be considered before substituting one for the other.

### Method A. Rate of Absorption

#### Procedure

18. The rate of water absorption of all sheet and plate insulating materials shall be determined in accordance with the Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup>

### Method B. Total Absorption at Saturation

#### Test Specimens

19. The test specimen shall be 3 by 1 in. by the thickness of the material for materials  $\frac{1}{8}$  in. in thickness or less. For materials over  $\frac{1}{8}$  in. in thickness the specimen shall be 3 by  $\frac{1}{8}$  in. by the thickness of the material. It shall be sawed or sheared from the sample so as to have smooth edges free from cracks. The cut edges of all test specimens shall be finished with No. 0 or finer sandpaper or emery cloth. The sawing and sandpapering operations should be slow enough so that the material is not heated appreciably. The thickness in inches to the nearest 0.001 in. shall be measured in the direction perpendicular to the faces of the original sample.

#### Conditioning

20. (a) To determine the total water absorbed at saturation, three specimens shall be tested individually, weighed, and then conditioned as described in Paragraphs (b) and (c).

(b) Materials whose water absorption value would be appreciably affected by temperature in the neighborhood of 110 C. shall be dried in an oven for 24 hr. at  $50 \pm 3$  C., cooled in a desiccator, and immediately reweighed.

(c) Specimens of materials such as phenolic laminated, and other products whose water absorption value has been shown not to be appreciably affected by temperatures up to 110 C. shall be dried in an oven for 1 hr. at 105 to 110 C.

#### Procedure

21. (a) The dried specimen shall be immersed in distilled water at 20 to 30 C. At the end of 24 hr. the specimen shall be removed from the water, the surface water wiped off with a dry cloth, and the specimen weighed immediately and then replaced in the water. The weighings shall be repeated every day for the first week and every week thereafter until the increase in weight, as shown by three consecutive weighings, averages less than 1 per cent of the total increase in weight, when the specimen shall be considered to be saturated.

(b) The difference between the saturated weight and the dry weight shall be considered as the water absorbed at saturation.

#### Report

22. The report shall include the test results for each of the three specimens as follows:

(1) The original thickness of material measured to the nearest 0.001 in.,

(2) The percentage loss in weight on drying, calculated on the original weight, and

(3) The percentage of water absorbed at saturation, calculated on the dry weight.

#### VOLATILE MATTER

##### Test Specimens

23. The test specimens shall conform

to the requirements specified in Section 19.

#### Procedure

24. To determine the volatile matter, three specimens shall be tested individually as follows: The specimen shall be weighed, dried in an oven at 105 to 110 C. until the decrease in weight as shown by two consecutive weighings at least 24 hr. apart is less than 1 per cent of the total decrease in weight. The difference between the original weight and the dry weight shall be recorded as the volatile matter present in the specimen as received (this includes in addition to water any volatile solvent left during manufacture). The percentage of volatile matter shall be based on the weight of the dry specimen.

#### Report

25. The report shall include the test results for each of the three specimens and the average values as follows:

(1) The original thickness of the material measured to the nearest 0.001 in., and

(2) The percentage of volatile matter, based on the weight of the dry specimen.

#### DIELECTRIC STRENGTH

##### Dielectric Strength

26. (a) Except as specified below in Sections 27 to 30, the dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149).<sup>3</sup>

(b) Tests shall be made transverse or parallel to the flat sides, or both, depending upon whether the stress on the material when in use is to be transverse or parallel to the flat sides, or both.

##### Electrodes and Test Specimens

27. (a) In the transverse direction of the specimen, the tests shall be made

between standard 2-in. electrodes (Note 1).

NOTE.—Surface creepage and flashover at the higher voltages may be prevented by pouring around the electrodes any high-grade insulating compound which will form a good bond with the material and with the electrodes. Sealing wax and phenolic resin varnish have been successfully used under certain conditions. In applying this protective compound, care shall be taken to prevent voids under the edges of the electrodes.

(b) For tests parallel to the flat sides (parallel with laminations in laminated sheets) the test specimens shall be  $\frac{1}{2}$  in. in width by 1 in. in length by the thickness of the material. Minimum thickness of material shall be  $\frac{1}{8}$  in. A hole shall be drilled in the approximate center of the 1-in. length in a direction parallel with the flat sides, to a depth of  $\frac{7}{16}$  in., leaving a thickness of  $\frac{1}{16}$  in. to be tested. A snug fitting metal pin electrode, with the end ground to conform with the shape of the drill used, shall be inserted in the hole. The specimen shall be placed on a flat metal plate, which shall be at least  $1\frac{1}{2}$  in. in diameter. This plate shall serve as the lower electrode. Thus, in effect, the material shall be tested parallel with the flat sides in a point-plane gap. The diameter of the hole shall be as shown in the following table:

Nominal Thickness of Sheets, in.	Nominal Hole Diameter for Pin Electrode, in.
$\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	$\frac{7}{16}$
Over $\frac{1}{2}$ .....	$\frac{1}{2}$

### Conditioning

28. (a) In the case of materials known to be affected by humidity, conditioning prior to dielectric strength test shall be carried out in accordance with the material specification applying to the material to be tested.

(b) When materials are to be used at other than room temperature, their di-

electric strength characteristic shall be determined over the operating range of temperature. Prior to test, specimens shall be exposed to each test temperature in a suitable temperature-control chamber for a period in minutes equal to one-half the thickness of the specimen in mils.

### Surrounding Medium

29. All tests shall be made under oil, maintained at the same temperature as the test specimen.

### Procedure

30. (a) Tests shall be made by either the short-time method or the step-by-step method.

(b) In tests made by the short-time method, voltage shall be increased at the rate of 0.5 kv. per sec.

(c) In tests made by the step-by-step method, the voltage at each step shall be applied for 1 min. The voltage shall be increased in increments as follows:

Breakdown Voltage by Short-Time Method	Increment of Increase of Test Voltage
25 kv. or less.....	1.0 kv.
Over 25 to 50 kv., incl.....	2.0 kv.
Over 50 to 100 kv., incl.....	5.0 kv.
Over 100 kv.....	10.0 kv.

(d) At least five tests shall be made at each test temperature in the short-time method, and at least three tests in the step-by-step method. When the range of test temperatures is considerable, tests should be made at not less than four temperatures, if a curve of dielectric strength against temperature is desired.

NOTE 1.—The thickness of the section shall be measured prior to breakdown. This can be done conveniently by measuring the length of the electrode, then the combined length of the specimen and electrode with the electrode inserted in place.

NOTE 2.—For sheets with exceptionally high dielectric strength, surface flashover may occur. Such cases should be noted in the report.

## PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT

### Procedure

31. (a) The phase difference and dielectric constant shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

(b) The phase difference and dielectric constant parallel with laminations shall be determined in accordance with the Tentative Method of Test for Power Factor and Dielectric Constant Parallel with Laminations of Laminated Sheet and Plate Insulating Materials (A.S.T.M. Designation: D 669).<sup>3</sup>

### BONDING STRENGTH

#### *For Laminated Materials $\frac{3}{16}$ in. or Over in Thickness*

NOTE.—The bonding strength value obtained depends upon the thickness of the specimen. Only specimens of the same thickness should be used for comparison purposes, preferably  $\frac{1}{4}$  in. in thickness or machined from a greater thickness to  $0.500 \pm 0.005$  in. in thickness.

### Apparatus

32. Any universal testing machine may be used, provided it is accurate to 1 per cent of the lowest load to be applied. The machine shall be fitted with a head containing a 10-mm. steel ball.

### Test Specimens

33. Test specimens shall be 1 in. square, smoothly and accurately cut on a smooth saw.

### Procedure

34. (a) Five specimens shall be tested. These shall be placed on edge and the steel ball shall be accurately centered between the edges and ends of the specimen.

(b) *Speed of Testing.*—The crosshead speed of the testing machine shall be

such that the load can be accurately weighed, but shall not exceed 0.050 in. per min. when the machine is running idle.

### Report

35. The report shall include the following:

- (1) The thickness of the material,
- (2) The load, expressed in pounds or kilograms, required to split the specimen, and
- (3) The room temperature.

### ROCKWELL HARDNESS

### Apparatus

36. The Rockwell hardness tester shall be used, conforming to the requirements of the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18).<sup>6</sup>

### Test Specimens

37. The specimen shall have a minimum thickness of  $\frac{1}{4}$  in. The specimen may be composed of a pile-up of several pieces of the material of the same thickness, provided that precaution is taken that the surfaces of the pieces are in contact and not held apart by burrs from saw cuts, etc. The specimen shall be 1 in. square if cut from sheet stock, or at least 1 sq. in. in area from other shapes.

### Procedure

38. (a) The procedure described in A.S.T.M. Methods E 18 shall be followed. The adjustment of speed of load application and the time of application of the major load are of great importance. The dash pot on the Rockwell tester shall be so adjusted that the operating handle completes its travel in from 4 to 5 sec. with no specimen on the machine and with the machine set up

<sup>6</sup> 1946 Book of A.S.T.M. Standards, Parts I-A and I-B.



to apply a major load of 100 kg. When so adjusted the period taken for mechanism to come to a stop with the specimen in place will vary from about 5 to 15 sec. depending upon particular specimen, the penetrator, and load used. In order to eliminate errors due to "creeping," and to the possibility of weight arm descending far enough to rest on the lifting link or pin (Note) instead of on the specimen, the major load shall be removed from the specimen within 7 to 8 sec. after the handle has been tripped, time being determined by a stopwatch. Readings shall be taken on either the M or R scale. The M scale requires a  $\frac{1}{4}$ -in. ball penetrator, a minor load of 10 kg., a major load of 100 kg., and the reading taken on the red scale. The R scale requires a  $\frac{1}{2}$ -in. ball penetrator, a minor load of 10 kg., a major load of 60 kg., and readings taken on the red scale. The Rockwell reading shall be taken to the maximum nearest full scale division reached within 45 sec. after removing the major load.

NOTE.—If the arm rests on the link or pin, the load or the penetrator or both should be changed.

(b) Unless otherwise specified in the individual methods or specifications for a particular material, the choice of scales to be used shall be determined as follows: If readings with the M scale show a total indentation of less than 150 scale divisions upon application of the major load, the M scale shall be used for the Rockwell test. If readings show a total indentation of 150 or more scale divisions the tester shall be changed to the R scale combination. The total indentation shall be determined by the number of divisions the pointer passes through during 7.5 sec. from the time of tripping the handle.

(c) All tests shall be made at a temperature of  $25 \pm 2$  C. ( $77 \pm 3.6$  F.).

(d) Care shall be taken that the test is not made so near the edge of the specimen that it will break out when the major load is applied.

(e) The specimen shall be supported in such a manner as to prevent errors due to overhang, when it does not balance itself on the anvil.

(f) The readings shall be taken on the red scale and shall be recorded as follows: Count the number of times the needle passes through zero on the red scale on the application of the major load. Subtract from this the number of times the needle passes through zero upon the removal of this load. If the difference is zero, the reading shall be recorded as over 100. If the difference is 1, the reading shall be recorded as between 0 and 100, and, if the difference is 2, the reading shall be recorded as negative.

#### Report

39. The report shall include the following:

(1) The Rockwell hardness number read on the red scale. The letter M or R shall precede the Rockwell hardness number depending on the combination which has been used in determining the hardness.

(2) The total thickness of the specimen, and

(3) The number of pieces in a specimen and their average thickness.

#### ASH

#### Test Specimen

40. The test specimen shall consist of 2 to 5 g. of finely divided particles, such as millings or filings, of the material.

#### Procedure

41. The test specimen shall be dried for 2 hr. at 105 to 110 C., weighed, then ignited to constant weight in a crucible,



and weighed. The percentage of ash shall be calculated, based on the weight of the dried specimen.

#### FLAMMABILITY

##### Purpose

42. The test for flammability (Note) is intended to evaluate the tendency of the material to burst into flame under the continuous application of heat.

NOTE.—This test is not suitable for determining the flammability of thermoplastic materials.

##### Apparatus

43. The apparatus shall consist of the following:

(a) *Shielding Box*.—A shielding box, made either of metal or wood, in which the test is conducted for the purpose of warding off air currents. This box shall be 12 by 24 in. by 16 in. in height and shall contain no front or top.

(b) *Resistance Wire*.—Seven feet of No. 30 B. & S. gage nichrome wire having a resistance of 6.75 ohms per foot plus or minus 5 per cent.

(c) An electrical connection for alternating or direct current of 110 v. Where necessary, the voltage may be brought to this value with a rheostat.

(d) A stop watch, voltmeter, and stand clamp.

##### Test Specimens

44. The test specimen shall be smooth-sawed from sheets to dimensions of  $\frac{1}{8} \pm 0.005$  in. by  $1 \pm 0.005$  in. by 8 in. The edges of the specimen shall be notched by a milling machine. The notches shall be rectangular,  $\frac{1}{32}$  in. in depth and 0.015 in. in width, and shall be spaced 10 notches to the inch. Binding posts shall be fastened at each end of

the specimen. The resistance wire shall be wound firmly about the specimen into the notches, and the ends shall be fastened to the binding posts.

##### Conditioning

45. The test specimens shall be conditioned for 24 hr. at 105 C. before test followed by cooling to room temperature in a desiccator. Tests shall be made at room temperature maintained at  $25 \pm 8$  C.

##### Procedure

46. Five specimens shall be tested. The shielding box shall be placed within a hood. The specimens shall be mounted on the stand clamp in a horizontal position with the laminations in a vertical position inside the box. The front of the hood shall be lowered to within 2 in. of the bottom. The starting switch shall be snapped on and at that instant the stop watch started. The voltage shall be adjusted to that specified and when the sample bursts into flame the watch shall be stopped and the time interval from the beginning of the test shall be recorded.

##### Report

47. The report shall state the number of seconds required for the specimen to burst into flame.

##### Coefficient of Linear Thermal Expansion

48. The coefficient of linear thermal expansion of all sheet and plate insulating materials shall be determined in accordance with the Standard Method of Test for Coefficient of Linear Thermal Expansion of Plastics (A.S.T.M. Designation: D 696).<sup>3</sup>

# *Standard Methods of Test for*

## THICKNESS OF SOLID ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 374 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 374; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover procedures for determining the thickness of solid insulating materials, except rubber insulating tape and friction tape for electrical purposes. Three alternative procedures are described, as follows:

*Method A* makes use of an adjusted ratchet micrometer together with a definite manipulative procedure by which the pressure exerted on the specimen is controlled.

*Method B*, known as the "feel" method, makes use of a machinist's micrometer constructed without a ratchet, pressure on the specimen being controlled by stopping closure of the micrometer when resistance to movement of the specimen between the instrument surfaces is first observed.

*Method C*, makes use of a dead weight dial micrometer, which is constructed so that measurements made with it are practically the same as those made with the method A ratchet micrometer.

Method A and method C instruments may be used interchangeably.

NOTE.—For determining the thickness of rubber insulating tape and friction tape for electrical purposes reference should be made to the Tentative Specifications for Rubber Insulating Tape (A.S.T.M. Designation: D 119)<sup>3</sup> and for Friction Tape for General Use for Electrical Purposes (A.S.T.M. Designation: D 69)<sup>3</sup> of the American Society for Testing Materials.

### Precision of Methods

2. (a) Methods A and C are preferred as reference standard methods for use in cases of dispute. They are also preferred for the measurement of compressible materials such as untreated paper and fabrics, as well as for the measurement of rigid materials. The maximum error (instrumental plus manipulative) of methods A and C is of the order of 0.0003 in.

(b) Method B may be used on rigid materials or on yielding materials where it is necessary to measure the specimen with practically no compression or deformation. The maximum error of method B is approximately 0.0005 in., except on unusually compressible ma-

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1933 to 1942, being revised in 1934, 1936, and 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

materials, where the error will be somewhat greater.

#### METHOD A. MACHINIST'S MICROMETER WITH RATCHET

##### Apparatus

3. The instrument used for determining thickness by Method A shall be a 1-in. machinist's type micrometer without a locking device. It shall be constructed with a vernier reading to 0.1 mil (0.0001 in.) and with a ratchet or similar mechanism for controlling measuring pressure, and shall have anvil and spindle surfaces  $0.250 \pm 0.001$  in. in diameter. The instrument shall conform to the requirements for flatness and parallelism of micrometer surfaces, zero reading, wear of micrometer screw, permissible micrometer screw error, and ratchet pressure specified in Sections 11 to 16. The micrometer shall be tested and calibrated periodically for conformity to these requirements.

##### Procedure

4. (a) Before starting measurements of thickness the micrometer shall be closed on the specimen at a location outside the area to be measured. The micrometer shall then be opened not more than 4 or 5 mils (0.004 or 0.005 in.) and then moved into the area selected for measurement. Using the ratchet, the micrometer surfaces shall be closed so slowly on the specimen that the mil scale divisions may be easily counted as they move past the reference mark, or at a rate of about 2 mils (0.002 in.) per second. The closing motion shall be continued at the same rate until the ratchet has clicked three times, and then the thickness shall be read by means of the vernier.

(b) In moving from one measurement location to another this operation shall be repeated, never opening the micrometer more than 4 or 5 mils (0.004 or

0.005 in.) more than the specimen thickness.

(c) In making a measurement, all points on the peripheries of the micrometer surfaces shall be at least  $\frac{1}{4}$  in. from the edges of the specimen.

#### METHOD B. MACHINIST'S MICROMETER WITHOUT RATCHET

##### Apparatus

5. The instrument used for determining thickness by Method B shall be a 1-in. machinist's type micrometer without a locking device. It shall be constructed with a vernier reading to 0.1 mil (0.0001 in.) and with anvil and spindle surfaces  $0.250 \pm 0.001$  in. in diameter. The instrument shall conform to the requirements for flatness and parallelism of micrometer surfaces, zero reading, and permissible micrometer screw error specified in Sections 11 to 13 and 15. The micrometer shall be tested and calibrated periodically for conformity to these requirements.

##### Procedure

6. (a) In the determination of thickness the micrometer shall be slowly closed on the specimen until contact is made without appreciable distortion of the specimen. The criterion of contact is the initial development of frictional resistance to movement of the specimen between the micrometer surfaces. The thickness shall then be read by means of the vernier.

(b) In moving from one measurement location to another this operation shall be repeated, never opening the micrometer more than 4 or 5 mils (0.004 or 0.005 in.) more than the specimen thickness.

(c) In making a measurement, all points on the peripheries of the micrometer surfaces shall be at least  $\frac{1}{4}$  in. from the edges of the specimen.

## METHOD C. DEAD WEIGHT DIAL MICROMETER

### Apparatus

7. (a) The micrometer shall be a dead weight dial type micrometer, having two ground and lapped circular surfaces, each  $0.250 \pm 0.001$  in. in diameter, and shall have a capacity of not less than 0.170 in.

(b) The surfaces shall be parallel to within 0.0001 in. and shall move on an axis perpendicular to themselves.

(c) The pressure exerted on the specimen shall be within the limits of 23 and 27 psi.

(d) The dial spindle shall be vertical and the dial shall be at least 2 in. in diameter. It shall be continuously graduated to read directly to 0.0001 in. and shall be equipped with a telltale hand, recording the number of complete revolutions of the large hand. The dial indicator mechanism shall be full jeweled.

(e) The micrometer shall be capable of repeating its readings to 0.00005 in. at zero setting or on a steel gage block.

(f) Measurements made on standard steel gages shall be within the following tolerances:

Intervals, in.	Permissible Deviation of Reading from Actual Thickness of Standard Steel Gage, in.
0 to 0.01.....	0.0001
Over 0.01.....	0.0005

(g) The deviations for the parts of the scale corresponding to the paper thickness measured, shall be applied as corrections to the thickness reading.

(h) The frame of the micrometer shall be so rigid that a load of 3 lb. applied to the dial housing, out of contact with either the weight or the presser foot spindle, will produce a deflection of the frame not greater than 0.0001 in., as indicated on the micrometer dial.

### Procedure

8. (a) The micrometer shall be placed on a solid, level table, free from excessive vibration. The specimen shall be placed between the micrometer surfaces and the presser foot lowered on the specimen at a location outside the area to be measured. The presser foot shall then be raised a distance of 0.3 to 4.0 mils, the specimen moved to the measurement position and the presser foot dropped onto the specimen.

NOTE.—The procedure described in Paragraph (a) minimizes small errors present when the presser foot is lowered slowly on the specimen. Care should be taken not to raise the presser foot more than 0.4 mil above the position of rest on the specimen surface.

(b) In making a measurement, all points on the peripheries of the micrometer surfaces shall be at least  $\frac{1}{4}$  in. from the edges of the specimen.

(c) For each succeeding measurement, the presser foot shall be raised 0.3 to 0.4 mils, the specimen moved to the next measurement location and the presser foot dropped.

(d) When compressible papers or fabrics are measured, a slight settling of the presser foot occurs. To minimize the errors produced by this effect, the reading of the dial indication should be deferred until the presser foot has been supported by the specimen for at least 2 sec., or until the micrometer hand becomes stationary.

### CALIBRATION OF MICROMETERS

#### Cleaning Surfaces of Machinist's and Dial Micrometers

9. Before and during instrument calibration and thickness measurements, the micrometer surfaces shall be maintained in a clean condition by lightly closing them on a clean sheet of smooth bond paper and moving the paper be-

tween the surfaces. To minimize the danger of the presence of lint, the edge of the paper should not be pulled between the surfaces.

#### Calibration of Machinist's Micrometers

10. (a) *Method A Micrometer.*—In the calibration of controlled pressure micrometers used in Method A, the micrometer shall be closed on the gage or calibrating device and then opened 4 or 5 mils (0.004 or 0.005 in.). Using the ratchet, the micrometer shall be again closed so slowly on the calibrating device that the mil scale divisions may be easily counted as they move past the reference mark, or at the rate of about 2 mils (0.002 in.) per sec. The closing motion shall be continued at the same rate until the ratchet has clicked three times, when the reading shall be taken.

(b) *Method B Micrometer.*—In the calibration of micrometers used in Method B the micrometer shall be slowly closed on the gage or calibrating device until contact of the surfaces and gage is made. The criterion of contact is the initial development of frictional resistance to movement of the gage device between the micrometer surfaces.

#### Flatness of Surfaces of Machinist's Micrometers

11. The anvil and spindle surfaces of the machinist's micrometer shall be flat to within 0.00005 in. The flatness may be determined by use of an optical flat. After cleaning the surfaces of the flat and the micrometer (Section 9) the latter shall be closed on the flat as described in Section 10 (a) or (b). When illuminated by diffused daylight, interference bands are formed between the surfaces of the flat and those of the micrometer. The location, shape, and number of these bands indicates the deviation from flatness in increments of half the average

of the wave lengths of white light, which is taken as 0.00001 in.

A *flat surface* forms straight, parallel and equidistant fringes.

A *grooved surface* forms straight parallel fringes at unequal intervals. The estimated maximum displacement of any line from its normal position, where all lines would be equidistant, is a measure of deviation from flatness.

A *symmetrical concave or convex surface* forms concentric circular fringes, and their number is a measure of deviation from flatness.

An *unsymmetrical concave or convex surface* forms a series of curved fringes, cutting the periphery of the micrometer surface. The number of fringes cut by a straight line connecting the termini of any fringe is a measure of the deviation from flatness.

#### Parallelism of Surfaces of Machinist's Micrometers

12. The anvil and spindle surfaces of the machinist's micrometer shall be parallel to each other to less than 0.0001 in. when tested with a pair of screw-thread-pitch wires or with a pair of  $\frac{1}{4}$ -in. nominal diameter plug gages. The diameters of the screw-thread-pitch wires or the plug gages, accurate to 0.00002 in., shall differ by an amount approximately equal to the axial movement of the spindle when rotated through 180 deg. (12.5 mils). The micrometer shall be closed on the wires or on the plug gages according to the procedure described in Section 10 (a) or (b). Observations made with either wire or with either plug gage placed at any location between the surfaces shall show differences of less than 0.0001 in.

#### Zero Reading of Machinist's Micrometers

13. The position of the anvil shall be such that a zero reading is obtained when the machinist's micrometer is

closed on the anvil as described in Section 10 (a) or (b). Ten trials shall give ten readings of zero. The condition of zero reading is satisfied when examinations with a low-power magnifying glass show that at least two-thirds of the widths of the zero graduation on the barrel and that of the reference mark coincide with each other.

#### **Wear of Machinist's Micrometer Screw**

14. The device for compensating for wear of the micrometer screw shall be adjusted so that the spindle has no perceptible lateral or longitudinal looseness, and yet may be rotated with a torque load of not more than  $\frac{1}{4}$  in-oz.

#### **Permissible Error in Machinist's Micrometer Screw**

15. The micrometer screw error, after zero adjustment is made, shall be checked at 2, 5, and 10 mils, and at intervals of 100 mils over the remaining graduated scale. For checks up to and including a thickness of 10 mils, selected gage blades the thicknesses of which are known to plus or minus 0.00002 in. shall be used. Checks at values greater than 10 mils shall be made with standard gage blocks. At each value checked, ten readings shall be taken and the arithmetic mean of these ten readings shall not differ from the thickness of the gage used by more than 0.1 mil (0.0001 in.). Manipulation of the instrument in these checks shall be in accordance with Section 10 (a) or (b).

#### **Ratchet Pressure, Method A Machinist's Micrometer**

16. The ratchet shall be so adjusted that a pressure of not more than 27 nor less than 23 psi. is developed when the spindle surface is contacted with a polished steel surface as described in Section 10 (a).

#### **Calibration of Dial Micrometer**

17. (a) In making these calibration measurements, the presser foot shall be raised from 0.3 to 0.4 mils above the position of contact with the steel ball or gage. After dropping the presser foot from the elevated position, the thickness reading shall be observed.

(b) *Parallelism of Surfaces.*—A hardened steel ball about  $\frac{1}{16}$  in. in diameter, fixed firmly in a flat metal handle about  $\frac{1}{32}$  in. in thickness, shall be measured at several locations on the micrometer surfaces, and the maximum variations of readings noted.

(c) *Accuracy of Scale Divisions.*—The instrument shall be set at zero and standard gages, the thickness of which is known to within 0.00001 in., shall be measured.

(d) *Pressure.*—The pressure applied to the presser foot spindle and weight necessary to move the pointer upward from the zero position shall not be greater than 650 g. The pressure applied to the presser foot spindle and weight necessary to just prevent movement of the pointer from a positive toward a lower reading shall not be less than 500 g.

# Standard Methods of

## TESTING LAMINATED TUBES USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 348 - 46

ADOPTED, 1939; REVISED, 1942, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 348; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover the procedures for testing laminated tubes to be used as electrical insulation.

### Conditioning

2. In order to eliminate the effects of previous history of humidity exposure and to obtain reproducible results (Note 1), the test specimens of laminated tubes shall be given a conditioning treatment for physical tests (Note 2) as follows:

(a) *Diametrical Compressive Strength.*—The machined specimens shall be conditioned prior to test by drying in an air-circulated oven at  $50 \pm 3$  C. for 48 hr., followed by cooling to room temperature in a desiccator. All specimens shall be tested at room temperature maintained at  $25 \pm 8$  C.

(b) *Tensile and Axial Compressive Strength, Density.*—No conditioning of specimens is required except in case of dispute, in which case the specimens shall be conditioned for 48 hr. at  $50 \pm 3$  C. In either case all specimens

shall be tested at room temperature maintained at  $25 \pm 8$  C.

NOTE 1.—Conditioning of specimens may be undertaken: (a) for the purpose of bringing the material into equilibrium with normal or average room conditions of 25 C. and 40 per cent relative humidity; (b) simply to obtain reproducible results, irrespective of previous history of exposure; or (c) to subject the material to abnormal conditions of temperature or humidity in order to predict its service behavior.

The conditions given here to obtain reproducible results may give physical values somewhat higher or somewhat lower than values under equilibrium at normal conditions, depending upon the particular material and test. To insure substantial equilibrium under normal conditions of humidity and temperature, however, will require from 20 to 100 days or more depending upon thickness and type of material and its previous history. Consequently, conditioning for reproducibility must of necessity be used for general purchase specifications and product control tests.

NOTE 2.—Conditioning of specimens for electrical tests is also necessary to obtain consistent results. The procedure for such conditioning is under consideration by the committee. In order to secure comparative results, specimens should be conditioned at the same temperature and humidity.

### Dimensional Measurements

3. Dimensional measurements of tubes shall be made in accordance with the Standard Methods of Measuring Dimen-

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1932 to 1939, being revised in 1932, 1934, 1935, and 1938.



sions of Rigid Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 668).<sup>3</sup>

### TENSILE STRENGTH

#### Apparatus

4. Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied. Jaws which tighten under load, such as wedge-grip jaws, shall be used with the specimen properly aligned. Steel or brass plugs,  $3\frac{1}{2}$  in. in length and of a suitable diameter, with edges rounded to  $\frac{1}{16}$ -in. radius will be required. The plugs shall fit the specimen within 0.010 in. They can conveniently be located in the tube by separating and supporting them on a metal rod, the lower end of which is screwed into the lower plug and the upper end of which rests in a loose socket as shown in Fig. 1.

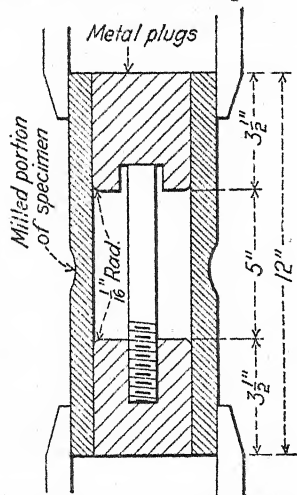


FIG. 1.—Metal Plugs Used for Tension Test Showing Location of Plugs in Test Specimen.

#### Test Specimens

5. The test specimens shall be 12 in. in length. When the wall thickness of tubing is  $\frac{1}{8}$  in. or over, a groove 1 in. in width and  $\frac{1}{2}$  in. in depth with corners rounded to a radius of 3 in. shall be made around the tube at the center.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

#### Procedure

6. (a) Five specimens shall be tested. The average inside and outside diameters determined from at least two measurements 90 deg. apart shall be measured at the groove to the nearest 0.001 in. and the cross-sectional area shall be calculated from these dimensions. The metal plugs shall be assembled with the tube as shown in Fig. 1. This assembly shall then be grasped in the V-notched jaws of the testing machine.

(b) *Speed of Testing.*—The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. when the machine is running idle.

#### Report

7. The report shall include the following:

(1) The average inside and outside diameters of the specimen expressed to the nearest 0.001 in., each determined from at least two measurements 90 deg. apart,

(2) The average outside diameter of the reduced section expressed to the nearest 0.001 in., if a groove was used,

(3) The full wall thickness of the specimen,

(4) The net area of the test section,

(5) The breaking load of each specimen in pounds,

(6) The tensile strength of each specimen in pounds per square inch, and

(7) The room temperature.

### COMPRESSIVE STRENGTH

#### Apparatus

8. Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top. The metal bearing plates shall be directly in contact with the test specimen.



### Test Specimens

9. Samples shall be tested as-received in specimen lengths of 1 in. Care shall be taken in cutting the test specimens for the axial test to have the ends of the specimens cut accurately at right angles to the axis of the tube. Specimens which are not cut squarely are likely to give low results on test.

### Procedure

10. (a) Five specimens shall be tested axially, with the load applied perpendicular to the faces or ends of the specimen, and five specimens shall be tested diametrically, with the load applied perpendicular to the tangent at point of application.

(b) *Speed of Testing.*—The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in per min. when the machine is running idle.

### Report

11. The report shall include the following:

(1) The average inside and outside diameters of the specimen expressed to the nearest 0.001 in., each determined from at least two measurements 90 deg. apart,

(2) The average wall thickness of the specimen expressed to the nearest 0.001 in.,

(3) The direction of application of the load,

(4) The load on each specimen at the first sign of rupture, in pounds, and

(5) The ultimate compressive strength in pounds per square inch calculated from the data obtained on the application of the load perpendicular to the face of the specimen.

### WATER ABSORPTION

#### Procedure

12. The rate of water absorption shall be determined in accordance with the Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.<sup>3</sup>

#### DENSITY

#### Test Specimens

13. Any suitable size specimen may be used. The specimen 1 in. in length used for the water absorption test (Section 12) will be found convenient.

#### Procedure

14. Two specimens shall be tested. Any suitable weight-difference apparatus for making readings in water and in air, or the weight and dimensions of the specimen, may be used as a basis for calculating the density.

#### Report

15. The report shall express the density in c. g. s. units.

### DIELECTRIC STRENGTH

#### Dielectric Strength

16. (a) Except as specified below in Sections 17 to 22, the dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149).<sup>3</sup>

(b) Tests shall be made transverse or parallel with the wall of the tube, or both, depending upon whether the stress on the tube when in use is to be transverse or parallel with the wall, or both.

#### Electrodes and Test Specimens

17. (a) *For Testing in Transverse Direction.*—The inner electrode shall consist of a brass rod, 3 in. in length; with edges rounded to a  $\frac{1}{4}$ -in. radius, and of such diameter that it fits snugly inside

the tube to be tested. The outer electrode shall consist of a strip of metal foil  $2\frac{1}{2}$  in. in width and long enough to extend around the circumference of the tube. The test specimen shall be at least 12 in. in length.

(b) *For Testing Parallel with Laminations.*—The test specimens shall be  $\frac{1}{2}$  in. in length. A hole shall be drilled into one end of the test specimen in the approximate center of the wall parallel with the major axis of the tube to a depth of  $\frac{7}{16}$  in. leaving a thickness of  $\frac{1}{16}$  in. to be tested. A snug-fitting metal-pin electrode, with the end ground to conform with the shape of the drill used, shall be inserted in the hole. The specimen shall be placed on a flat metal plate having a diameter at least  $\frac{1}{2}$  in. greater than that of the specimen. This plate shall serve as the lower electrode. Thus, in effect, the material shall be tested parallel with lamination in a point-plane gap. The diameter of the hole shall be as shown in the following table:

Nominal Wall Thickness of Tubes, in.	Nominal Hole Diameter for Pin Electrode, in.
$\frac{1}{8}$ to $\frac{1}{4}$ , incl. ....	$\frac{1}{16}$
Over $\frac{1}{4}$ .....	$\frac{3}{8}$

NOTE.—For tubes with exceptionally high dielectric strength, surface flashover may occur. Such cases shall be noted in the report.

### Conditioning

18. (a) Unless otherwise specified, all test specimens shall be conditioned for 48 hr. at  $50 \pm 3$  C. in a circulating air oven prior to testing. After removal from oven, specimens shall be permitted to cool to room temperature in a desiccator over anhydrous calcium chloride.

(b) In the case of tubes to be used at other than room temperature, the dielectric strength characteristics shall be determined over the operating range of temperature. Prior to test, specimens previously conditioned as described in

Paragraph (a) shall be exposed to each test temperature in a suitable temperature-control chamber for a period in minutes equal to one half the wall thickness of the specimen in mils.

### Surrounding Medium

19. The specimens shall be tested immersed in oil maintained at the temperature of the test specimen.

### Procedure

20. (a) Tests shall be made by either the short-time method or the step-by-step method.

(b) In tests made by the short-time method, the voltage shall be increased at the rate of 0.5 kv. per sec.

In tests made by the step-by-step method, the voltage at each step shall be applied for 1 min. The voltage shall be increased in increments as follows:

Breakdown Voltage by Short-Time Method	Increment of Increase of Test Voltage
25 kv. or less .....	1.0 kv.
Over 25 to 50 kv., incl. ....	2.0 kv.
Over 50 to 100 kv., incl. ....	5.0 kv.
Over 100 kv. ....	10.0 kv.

### Number of Tests

21. At least five tests shall be made at each temperature in the short-time method, and at least three tests in the step-by-step method. When the range of test temperatures is considerable, tests should be made at not less than five temperatures, if a curve of dielectric strength against temperature is desired.

### Report

22. The report shall include the following:

(1) A description of the material including name, type, grade, color, size, and name of the manufacturer.

(2) A statement of the direction of dielectric stress application whether transverse to or parallel with laminations.

(3) The conditioning treatment which the specimens have received.

(4) A statement of the procedure used, whether short-time method or step-by-step method.

(5) Nominal wall thickness of the tube in inches.

(6) The maximum, minimum, and average puncture voltage in kilovolts and volts per mil (Note).

(7) Duration of test, if step-by-step method, including the initially applied voltage in kilovolts.

(8) The temperature of the test specimen.

(9) The size and type of electrodes.

NOTE.—To calculate the volts per mil, the wall thickness in transverse tests on material undisturbed by breakdown but as near the point of breakdown as possible shall be used. For

tests parallel with laminations, the thickness of the section shall be measured prior to breakdown. This can be conveniently done by measuring the length of the electrode, then the combined length of the specimen and electrode with the electrode inserted in place

#### PHASE DIFFERENCE (POWER FACTOR) AND DIELECTRIC CONSTANT

##### Phase Difference and Dielectric Constant

23. The phase difference and dielectric constant shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup>

# *Standard Methods of*

## MEASURING DIMENSIONS OF RIGID TUBES USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 668 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 668; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover procedures for measuring the dimensions of all rigid tubes used in electrical insulation, the limitations imposed being those of the size range of the more common forms of measuring instruments used.

### Conditioning

2. In cases of disagreement between the seller and the purchaser because of dimensional changes resulting from changes in temperature and humidity, all specimens shall be conditioned for 48 hr. at  $25 \pm 1$  C. and  $50 \pm 2$  per cent relative humidity, and measured under these conditions. Measurements by both interested parties shall be made within a three-week period.

### Method of Measurement

3. All measurements shall be made in accordance with the procedures described in the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation:

D 374) of the American Society for Testing Materials,<sup>3</sup> as applicable. The micrometer or vernier calipers shall be slowly closed on the specimen until contact is made without appreciable distortion of the specimen. The criterion of contact is the initial development of frictional resistance to movement of the specimen between the micrometer or caliper surfaces.

### Length of Tubes 12 in. and Under in Length

4. (a) *Apparatus*.—A standard machinist's micrometer or vernier calipers of suitable size reading to 1 mil (0.001 in.) shall be used.

(b) *Test Specimens*.—Specimens shall consist of tubes cut to lengths of 12 in. and under.

(c) *Procedure*.—The length of the specimen shall be measured to the nearest 0.001 in. Four measurements shall be made at points 90 deg. apart around the circumference of the specimen.

(d) *Report*.—The average of the measurements taken shall be reported as the length of the tube.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1942 to 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

**Length of Tubes Over 12 in. in Length**

5. (a) *Apparatus*.—A machinist's precision steel tape or steel scale of suitable length graduated in  $\frac{1}{64}$  in. shall be used.

(b) *Test Specimens*.—Specimens shall consist of tubes over 12 in. in length, including full lengths.

(c) *Procedure*.—The length of the specimen shall be measured to the nearest  $\frac{1}{64}$  in. Four measurements shall be made at points 90 deg. apart around the circumference of the specimen.

(d) *Report*.—The average of the measurements taken shall be reported as the length of the tube.

**Outside Diameter**

6. (a) *Apparatus*.—A standard machinist's micrometer or vernier calipers reading to 1 mil (0.001 in.) shall be used (Note).

NOTE.—Where the number of tests to be made or the specifications covering the tube justify its use, an accurately calibrated "go-and-no-go" ring gage of suitable size may be used.

(b) *Test Specimens*.—Specimens shall consist of tubes 12 in. and under in outside diameter.

(c) *Procedure*.—The outside diameter of the specimen shall be measured to the nearest 0.001 in. Four measurements shall be made at points 45 deg. apart around the circumference at each end and at the middle of the specimen.

(d) *Report*.—The average of the 12 measurements taken shall be reported as the outside diameter of the tube.

**Variation in Outside Diameter**

7. (a) *Definition*.—Variation in outside diameter is defined as the greatest difference in measurements of the outside diameter.

(b) *Apparatus*.—A standard machinist's micrometer or vernier calipers reading to 1 mil (0.001 in.) shall be used.

(c) *Test Specimen*.—Specimens shall

consist of tubes 12 in. and under in outside diameter.

(d) *Procedure*.—The outside diameter of the specimen shall be measured to the nearest 0.001 in. A series of measurements shall be made around the circumference of the specimen at each end and at the center to determine the maximum and minimum values.

(e) *Report*.—The difference between the maximum and minimum values shall be reported as the variation in outside diameter. All measurements taken shall also be reported.

**Inside Diameter**

8. (a) *Apparatus*.—A machinist's inside micrometer or vernier calipers of suitable size reading to 1 mil (0.001 in.) shall preferably be used, although a combination of telescoping gage and standard machinist's outside micrometer may be used (Note).

NOTE.—Where the number of tests to be made or the specifications covering the tube justify its use, an accurately calibrated "go-and-no-go" plug gage of suitable size may be used.

(b) *Test Specimen*.—Specimens shall consist of tubes whose inside diameter is not over 12 in., nor under  $\frac{1}{8}$  in.

(c) *Procedure*.—The inside diameter of the specimen shall be measured to the nearest 0.001 in. Four measurements shall be made at points 45 deg. apart around the circumference at each end of the specimen.

(d) *Report*.—The average of the measurements taken shall be reported as the inside diameter of the tube.

**Wall Thickness**

9. (a) *Apparatus*.—A ball foot micrometer or suitable vernier calipers reading to 1 mil (0.001 in.) shall be used for the measurement of tubes  $\frac{5}{16}$  in. and over in inside diameter. A dial micrometer reading to 1 mil (0.001 in.)

shall be used for the measurement of tubes under  $\frac{5}{16}$  in. in inside diameter.

(b) *Test Specimens*.—Specimens shall consist of tubes of any length or diameter.

(c) *Procedure*.—The wall thickness shall be measured to the nearest 0.001 in.

For tubes  $\frac{5}{16}$  in. and over in inside diameter, four measurements shall be taken at points 90 deg. apart around the circumference at each end of the specimen.

For tubes under  $\frac{5}{16}$  in. in inside diameter, the wall thickness shall be measured by placing the specimen over a mandrel of slightly smaller diameter which is fixed in position at right angles relative to the spindle of the dial micrometer. A zero setting shall be noted with the spindle against the pin. Then the specimen shall be placed over the pin and readings taken at various points around the circumference of the specimen. At least four readings shall be taken.

(d) *Report*.—The average of the measurements taken shall be reported as the wall thickness of the tube.

#### Variations in Wall Thickness

10. (a) *Definition*.—Variation in wall thickness is defined as the difference between high or low measurements.

(b) *Apparatus*.—The apparatus shall be the same as specified in Section 9 (a).

(c) *Test Specimens*.—Specimens shall consist of tubes of any length or diameter.

(d) *Procedure*.—The wall thickness shall be measured to the nearest 0.001 in.

For tubes  $\frac{5}{16}$  in. and over in inside diameter, a series of measurements shall be made around each end of the specimen.

For tubes under  $\frac{5}{16}$  in. in inside diameter, the variation in wall thickness shall be measured by rotating the

specimen around the mandrel and following the general procedure described in Section 9 (c) for tubes of this size.

(e) *Report*.—The difference between the maximum and minimum values, shall be reported as the variation in wall thickness. All measurements taken shall also be reported.

#### Warp

11. (a) *Apparatus*.—A horizontal flat surface and a rigid bar with a vertically plane surface firmly fixed at right angles to the flat surface both being at least as long as the specimen shall be used. The height of the bar shall exceed half the diameter of the tube. Feeler gages of suitable thicknesses will also be required.

(b) *Test Specimens*.—Specimens shall consist of tubes of any length or diameter.

(c) *Procedure*.—The specimen shall be placed on the horizontal flat surface and rotated against the vertically plane surface of the rigid bar. The bar shall be firmly fastened to the horizontal flat surface. With the aid of feeler gages, the maximum distance of the nearest point of the tube from the vertically plane surface shall be measured to the nearest 0.001 in.

(d) *Report and Calculation*.—Warp or lack of straightness shall be reported as the maximum distance of any part of the tube from a straight edge which connects the ends of the specimen. The warp shall be calculated as follows:

$$W = \frac{D \times 100}{L}$$

where:

$W$  = percentage of warp,

$D$  = maximum deviation of tube from straight edge in inches, and

$L$  = length of tube in inches as determined in Sections 4 or 5.

In order to compare the relative warp-age for any length of tube, the warp shall be calculated as the percentage warp for a 36-in. length as follows:

$$C = \frac{W \times 36}{L}$$

where:

$C$  = percentage of warp calculated for a 36-in. length,

$W$  = percentage of warp, and

$L$  = length of tube in inches as determined in Sections 4 or 5.

*Standard Methods of*  
TESTING FLEXIBLE VARNISHED TUBING USED FOR  
ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 350 - 45

ADOPTED, 1943; REVISED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 350; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These methods cover the procedures for the testing of flexible varnished tubing and saturated sleeving (Note) used as electrical insulation for leads on electrical apparatus.

NOTE.—The term “varnished tubing” as used in these methods refers to braided cotton sleeving coated, or impregnated and coated, with varnish, lacquer, or a combination of lacquer and varnish, or similar coatings. The term “saturated sleeving” refers to braided cotton sleeving impregnated with varnish but the coating need not be continuous as it is for varnished tubing.

#### Sampling

2. (a) A sufficient number of the pieces of tubing to total not less than 1.83 m. (6 ft.) in length shall be selected from each 305 m. (1000 ft.) in the shipment, but in no case shall less than 1.83 m. (6 ft.) be taken from a shipment, and the pieces shall be selected in such a

manner as to be representative of the shipment.

(b) The number of specimens required for purpose of tests shall be cut from the pieces selected in accordance with Paragraph (a), and care shall be taken to select material which is free from abnormal defects such as blisters, wrinkles, cracks, etc.

#### Conditioning

3. The test specimens shall be conditioned for 48 hr. in air maintained at a temperature between 20 and 30 C. and at a relative humidity of  $50 \pm 2$  per cent. If a conditioning cabinet is used, specimens used for dielectric strength tests shall be subjected to test immediately upon withdrawal from the cabinet.

NOTE.—A saturated solution of sodium dichromate is a suitable humidifying agent for obtaining these limits when used under the conditions specified in Section 11 (b) of the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257) of the American Society for Testing Materials.<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1932 to 1943, being revised in 1935, 1937, 1938, 1939, and 1940.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



## DIMENSIONS

## Apparatus

4. (a) *Gage Rods*.—Standard gage rods with hemispherical ends shall be used. The standard gage rods shall be made of steel and shall have smooth surfaces. A set of two is required for each size of tubing. Each rod in a set shall have a diameter within plus or minus 0.0002 in. of the diameters listed as maximum and minimum in Table I.

TABLE I.—A.S.T.M. STANDARD SIZES FOR FLEXIBLE VARNISHED TUBING.

Size	Inside Diameter, in.		
	Maximum	Minimum	Nominal
1 in. ....	1.036	1.000	.....
$\frac{3}{4}$ in. ....	0.911	0.875	.....
$\frac{1}{2}$ in. ....	0.786	0.750	.....
$\frac{3}{8}$ in. ....	0.655	0.625	.....
$\frac{1}{4}$ in. ....	0.524	0.500	.....
$\frac{3}{16}$ in. ....	0.462	0.438	.....
$\frac{1}{8}$ in. ....	0.399	0.375	.....
No. 0.....	0.347	0.325	0.330
No. 1.....	0.311	0.289	0.294
No. 2.....	0.278	0.258	0.263
No. 3.....	0.249	0.229	0.234
No. 4.....	0.224	0.204	0.208
No. 5.....	0.198	0.182	0.186
No. 6.....	0.178	0.162	0.166
No. 7.....	0.158	0.144	0.148
No. 8.....	0.141	0.129	0.133
No. 9.....	0.124	0.114	0.118
No. 10.....	0.112	0.102	0.106
No. 11.....	0.101	0.091	0.095
No. 12.....	0.089	0.081	0.085
No. 13.....	0.080	0.072	0.076
No. 14.....	0.072	0.064	0.066
No. 15.....	0.067	0.057	0.059
No. 16.....	0.061	0.051	0.053
No. 17.....	0.054	0.045	0.047
No. 18.....	0.049	0.040	0.042
No. 19.....	0.044	0.036	0.038
No. 20.....	0.039	0.032	0.034

NOTE 1.—The minimum inside diameters prescribed in this table for sizes Nos. 0 to 20, inclusive, are the same as the diameters of copper wire for corresponding A.W.G. or B. & S. gage sizes.

NOTE 2.—Sizes Nos. 14, 16, 18, and 20 are standard and should be specified if possible. Sizes Nos. 15, 17, and 19 are special and may eventually be eliminated.

(b) *Micrometer*.—A 1-in. machinist's type micrometer with ratchet device and having anvil and spindle surfaces  $0.25 \pm 0.001$  in. in diameter shall be used.

## Test Specimens

5. Test specimens of any convenient length shall be cut from the samples obtained in accordance with Section 2.

## Procedure

6. (a) *Inside Diameter*.—The minimum gage rod for the size tubing under test shall pass into the specimen for a distance of 5 in. without expanding the wall of the tubing. If the rod has a fairly snug fit, then the specimen shall be considered as having an inside diameter equal to the diameter of the rod. If, however, the minimum gage rod fits loosely, then the maximum gage rod shall be inserted into the specimen. If the maximum gage rod passes freely into the specimen for a distance of 5 in. with a fairly snug fit or if it expands the wall of the specimen, then the tubing shall be considered to be of that size which falls within the limits of the maximum and minimum inside diameters as represented by the gage rods.

(b) *Wall Thickness*.—A standard gage rod which will pass freely into the tubing shall be inserted in the specimen. The micrometer shall then be applied over the specimen, and thickness measurements made as specified in Method A of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup> The average of five micrometer readings shall be obtained. The wall thickness shall be considered to be one-half the difference between the average micrometer reading and the diameter of the gage rod.

(c) *Outside Diameter*.—The outside diameter of the specimen shall be considered to be the sum of the estimated inside diameter and twice the wall thickness (Paragraph (b)). The inside diameter shall be estimated from the fit of the maximum and the minimum gage rods (Paragraph (a)).

## Report

7. The report shall include the following:

- (a) The size of the tubing,
- (b) The wall thickness in inches, and
- (c) The outside diameter in inches.

### DIELECTRIC STRENGTH

#### Dielectric Strength

8. The dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials,<sup>3</sup> except as specified in the following Sections 9 to 12.

#### Apparatus

9. (a) *Inner Electrode*.—A straight standard annealed copper wire shall be used as the inner electrode. The nearest A.w.g. size of wire shall be selected that will fit snugly without stretching the tubing. The wire shall be free of bends to avoid a binding action on the tubing as it is being slipped onto the wire.

(b) *Outer Electrode*.—Strips of metal foil shall be used as the outer electrode. The strips shall be 2.54 cm. (1 in.) in width and not more than 0.0005 in. in thickness.

NOTE.—The standard gage rods should never be used as the inner electrode. The arc which forms when the tubing punctures will invariably raise a pimple of melted metal on the surface of the rod, spoiling it for use as a standard.

#### Test Specimens

10. Test specimens of convenient length shall be cut from the samples selected in accordance with Section 2.

#### Procedure

11. (a) The tubing shall be placed on the inner electrode, the outer electrode consisting of strips of metal foil (Section 9 (b)) shall be tightly wrapped on the outside at a distance of not less

than 7.62 cm. (3 in.) apart and at least 7.62 cm. (3 in.) from the ends of the specimen. The first turn of the foil applied to the tubing shall be wiped snugly against the tubing. Two more turns of the foil shall then be wound over this first turn, leaving a free end of  $\frac{1}{2}$  in. to which the electrical connection shall be made during the test.

(b) The dielectric strength shall be determined by the short-time test. Voltage shall be increased at the rate of 0.5 kv. per sec. The tests shall be made in air.

(c) The dielectric strength shall be the average of ten breakdown voltages.

#### Report

12. The report shall include the following:

- (a) Total volts at each puncture,
- (b) The average, maximum, and minimum puncturing voltages,
- (c) The room temperature, and
- (d) The percentage relative humidity.

### AGING TEST

#### Test Specimens

13. Not less than ten test specimens, each 15.24 cm. (6 in.) in length shall be cut from the samples obtained in accordance with Section 2.

NOTE.—If the aging test is in excess of 120 hr., additional specimens will have to be cut.

#### Procedure

14. (a) The specimens shall be placed in a uniformly heated electric oven of the analytical type in which the temperature is maintained between 105 and 110 C. (221 and 230 F.). Two specimens shall be removed at the end of 24 hr. and every 24 hr. thereafter until failure occurs. After cooling to room temperature of approximately 20 C. (68 F.),

each specimen shall be bent through 180 deg. over a rod having a diameter as shown in the following table:

Size	Diameter of Test Rod, in.
1 to $\frac{3}{4}$ in., incl.....	$\frac{7}{16}$
$\frac{3}{4}$ to $\frac{1}{2}$ in., incl.....	$\frac{3}{8}$
Nos. 0 to 5, incl.....	$\frac{5}{16}$
Nos. 6 to 11, incl.....	$\frac{3}{16}$
No. 12 and smaller.....	$\frac{1}{8}$

(b) The number of hours of baking at which first cracking across the specimen occurs shall be noted and reported. Any side cracking occurring because of the flattening of the specimen on the rod shall be disregarded.

#### HEAT ENDURANCE

##### Apparatus

15. The apparatus for the heat endurance test shall be a thermometer and a uniformly heated oven. The oven shall have sufficient heating capacity to permit the desired temperature to be regained within 5 min. after closing the door subsequent to the introduction of specimens.

##### Test Specimens

16. At least three test specimens 9.2 cm. (4 in.) in length shall be cut from the samples selected in accordance with Section 2.

##### Procedure

17. A substantial cotton or linen string shall be tied around each specimen at mid-length and the specimens suspended horizontally in the oven maintained at a temperature between 425 and 450 F. As soon as the specimens have been mounted in the oven, the oven door shall be closed, and 15 min. later the specimens shall be removed and cooled to room temperature. The specimens shall then be examined for signs of softening, blistering, and flowing.

##### Report

18. The report shall include the following:

(a) Temperature of the oven to the nearest 2 F.,

(b) Length of time specimens were in the closed oven,

(c) Description of any signs of softening, blistering, or flowing.

#### RATE OF BURNING

##### Apparatus

19. The apparatus for the rate of burning test shall be a bunsen burner and a stop watch.

##### Test Specimens

20. At least three test specimens 9.16 cm. (4 in.) in length shall be cut from the samples obtained in accordance with Section 2.

##### Procedure

21. (a) A gage length of 2.54 cm. (1 in.) shall be marked on each test specimen approximately 1.27 cm. ( $\frac{1}{2}$  in.) from one end of the specimen.

(b) The end of the specimen on which the gage marks have been placed shall be inserted approximately 0.63 cm. ( $\frac{1}{4}$  in.) into the side of the flame of the bunsen burner with the lower side of the tubing approximately 1.27 cm. ( $\frac{1}{2}$  in.) above the top of the burner. The specimen shall be rotated in the flame to ignite it as uniformly as possible. The tubing shall then be removed from the flame and held vertically, the burning end uppermost, in still air. When the leading edge of the flame reaches the upper gage mark, the stop watch shall be started and the time in seconds for the leading edge of the flame to travel down the specimen to the lower gage mark shall be observed.

##### Report

22. The rate of burning for each specimen shall be reported.

## *Standard Methods of* TESTING ELECTRICAL INSULATING OILS<sup>1</sup>



A.S.T.M. Designation: D 117 - 43

ADOPTED, 1924; REVISED, 1927, 1931, 1933, 1936, 1940, 1942, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 117; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods<sup>2a</sup> of testing electrical insulating oils apply to mineral oil of petroleum origin for use in transformers, oil circuit breakers, and other electrical apparatus as an insulating or cooling medium, or both.

NOTE.<sup>3</sup>—For oils having a Saybolt Universal viscosity over 110 sec. at 100 F. (37.8 C.), modifications of certain test procedures are necessary. These modifications are under study and development by Committee D-9. A notation precedes each test procedure where such modification is necessary.

### SAMPLING

#### General

2. Samples shall be taken and handled in such a manner that the tests made upon them indicate the characteristics of the shipment. Some tests are greatly affected by the most minute traces of impurities. This is particularly the case with the sludge test and the dielectric strength test, the latter being affected by almost infinitesimal traces of

moisture. If these tests are to be made, it is important that the samples be handled with the utmost care to prevent such contamination. All tests are usually made on samples taken from drums. Sometimes dielectric strength tests are not made on samples taken from tank cars. Samples from tank cars are usually tested only for the physical and chemical properties that are not affected by *slight* amounts of moisture; care, however, shall be taken to prevent extraneous contamination of the samples as contamination might affect the sludge test. It is imperative, therefore, that the procedure and precautions for obtaining and transporting samples of the oil to be tested, as specified in Sections 3 to 8, inclusive, shall be observed.

#### Sampling Thiefs

3. (a) *General*.—Thiefs shall be used for withdrawing samples of oil from drums, tank cars, or other containers of 5-gal. capacity or larger in which shipment is made. Glass or metal thieves may be used.

(b) *For Drums*.—For sampling drums the thieves shall be so designed that they will reach within approximately  $\frac{1}{8}$  in. (about 3 mm.) of the bottom and should have a capacity of approximately 1 pt. or 1 qt. (0.5 or 1 liter).

<sup>1</sup> Under the standardization procedure of the Society these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials. In using these methods, reference should always be made to any pending revisions affecting the methods.

<sup>2a</sup> Prior to adoption as standard, these methods were published as tentative from 1921 to 1924.

<sup>3a</sup> A tentative revision of this standard has been issued in the form of the Tentative Methods of Testing Electrical Insulating Oils (D 117 - 46 T), see p. 600.

<sup>3</sup> Editorially revised in September, 1944.

NOTE.—A convenient and simple thief for sampling 50-gal. drum containers is shown in Fig. 1, which illustrates a sheet metal thief 36 in. in length,  $1\frac{1}{2}$  in. in diameter, with cone-shaped caps over the ends and having openings at the ends  $\frac{3}{8}$  in. in diameter. Three legs, equally spaced around the thief at the lower end long enough to hold the opening  $\frac{3}{8}$  in. from the bottom of the container, aid in securing a good representative sample. Two rings soldered to opposite sides of the tube at the upper end will be found convenient for holding the thief by slipping two fingers through them, leaving the thumb free to close the opening.

(c) *For Cans.*—For sampling cans of 5-gal. capacity or larger a thief, similar to that used for sampling drums but of

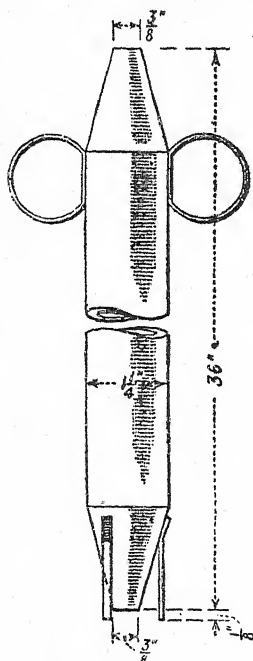


FIG. 1.—Thief for Sampling Drums.

proportionately smaller dimensions, shall be used. Cans of less than 5-gal. capacity shall be taken as a whole (see Section 7 (e)).

(d) *For Tank Cars.*—For sampling tank cars the thief shall be so designed that the sample may be obtained from within at least 0.5 in. of the bottom.

NOTE.—A thief suitable for sampling tank cars may be made from metal tubing and castings, machine-finished all over. A valve rod whose projecting stem strikes the bottom of the car, opens the valve automatically and simultaneously releases the air through the top. Such a thief is illustrated in Fig. 2.

#### Sample Containers

4. Tin cans, or cork-stoppered or glass-stoppered clear-glass bottles in cartons may be used as containers for the samples. The containers shall be clean and dry.

NOTE.—The clear-glass bottle offers the advantage that it may be examined to see that it

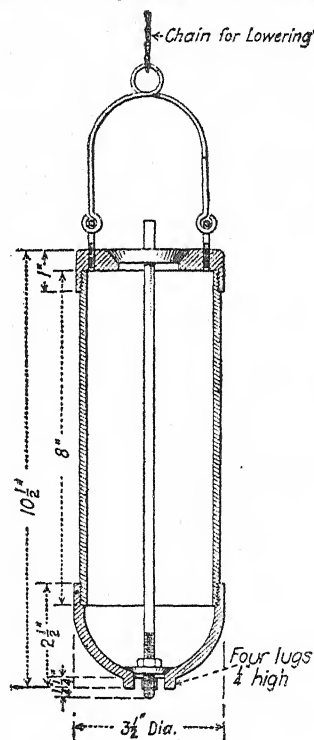


FIG. 2.—Thief for Sampling Tank Cars.

is clean. It also allows visual inspection of the oil before testing, permitting the detection of free water or solid impurities. Samples placed in glass bottles shall be kept in the dark if subsequent testing is to include color and sludge forming characteristics or neutralization value, as light produces changes in these properties.

For this reason cardboard cartons with covers shall be provided for each bottle into which the bottle with its sample of oil shall be placed immediately after sampling. Rubber stoppers shall never be used. If cork is used it shall be of good quality with a new cork for each sample. Contact of the oil with the cork may be prevented by wrapping some tin or aluminum foil around the cork before forcing it into the mouth of the bottle. If glass-stoppered bottles are used, precaution shall be taken to insure that perfectly fitting stoppers are secured. A tin can fitted with a screw cap protected with a cork disk faced with tin or aluminum will endure harder usage. If tin cans are to be employed, only those having seams soldered on the exterior surfaces shall be used.

### Cleaning of Sampling Apparatus

5. (a) *General*.—All thieves and sample containers shall be cleaned thoroughly before using, special care being taken that no lint or other fibrous material remains in or on them. Care shall be taken to insure the complete removal of all traces of soldering flux from new thieves and new cans.

NOTE 1.—Thieves and containers that are not chemically clean and dry are likely to contaminate the samples and cause misleading results on some of the tests.

NOTE 2.—To insure the greatest ease in removing traces of soldering flux, a flux of rosin in a suitable solvent is recommended for soldering all seams. Such a flux is easily removed with gasoline, whereas, many other fluxes are very hard to remove. Minute traces of flux may contaminate the sample so that the results obtained on dielectric strength tests and various sludge tests may be erroneous.

(b) *Thiefs*.—Thiefs, before using, shall be cleaned by rinsing with oil-free gasoline and dried in the hot cabinet described in Section 6 (a). The best procedure is to rinse them after the sampling has been finished and then place them in the hot cabinet, as in this way they will be ready for use without further cleaning when the next shipment is to be sampled.

(c) *Sample Containers*.—Sample containers, before using, shall be rinsed with

oil-free gasoline and dried. They shall then be washed with strong soapsuds, rinsed thoroughly with distilled water and then dried by passing a current of warm air through the containers, after which they shall be immediately placed in the hot cabinet described in Section 6 (a). A similar washing will be sufficient for re-use of the containers that have held new oil, but those which have contained used oil shall be washed with both gasoline and benzol before being washed with soapsuds.

### Storage of Sampling Apparatus

6. (a) *General*.—When not in use all thieves and sample containers shall be kept in a hot, dry, dust-free cabinet or compartment at a temperature not less than 100 F. (37.8 C.).

(b) *Thiefs*.—Thiefs shall be kept at all times in a vertical position in the hot cabinet in a rack having a suitable drainage receptacle at the base.

(c) *Sample Containers*.—Containers for samples, after washing and drying as described in Section 5 (c), shall be kept unstoppered while in the hot cabinet but shall be closed immediately on removal. When containers must be prepared in advance and cannot be kept in the hot cabinet until approximately the time for the sample to be placed therein (as for example, when bottle containers are prepared to be sent to a distant sampling point) the same procedure of washing and drying shall be followed as described in Section 5 (c). In such instances, after the bottles are corked or stoppered and removed from the hot cabinet, the corks or stoppers shall be capped with paper which is tied securely on the neck, and then the capped corks or stoppers shall be sealed by dipping into melted paraffin wax so as to cover all of the paper cap and part of the neck. If this procedure is followed, care shall be taken in removing the paraffined caps

preparatory to filling the bottles with samples, that no paraffin contaminates the sample. During the preparation of the containers and subsequent handling of them when introducing the sample, care shall be taken that the operator's fingers do not touch the lip of the container.

#### Procedure for Sampling New Oils

7. (a) *General*.—A sufficient number of samples shall be taken to cover the requirements of the tests. It is recommended that 1 qt. of oil be taken as a sample if complete physical and chemical tests are to be made and at least 1 pt. if only the dielectric strength tests are to be made.

(b) Drums, tank cars, or other containers of oil shall not be sampled until the oil is at least as warm as the surrounding air, because cold oil may condense enough moisture from a humid atmosphere to affect seriously its insulating properties. Samples shall, of course, never be taken in the rain and precautions shall be taken during sampling to prevent contamination such as that which may occur from wind-blown dust or other sources.

(c) Samples of oil from drums and cans shall be taken only after the oil has remained undisturbed for at least 8 hr.

(d) Drums and barrels of oil to be sampled shall be lined up, preferably on their sides with their bungs up. The bungs shall be unsealed, removed, and laid with the oily sides up beside the bungholes. The top hole of the thief shall be closed with the thumb and the thief thrust about 1 ft. (30 cm.) into the oil. The thumb shall then be removed, allowing the oil to flow into the thief. The upper end of the thief shall again be closed with the thumb and the thief withdrawn. The oil remaining in the thief shall be used for rinsing out the thief by holding the thief nearly horizon-

tal and turning so that the oil comes in contact with that part of the inside surface of the thief which is immersed when taking the final sample. During this rinsing of the thief, care shall be taken to avoid handling any portion of the thief that is subsequently immersed in the oil to be sampled as moisture from the fingers or hands may be sufficient to cause enough contamination of the oil to cause a low value in the dielectric strength test. The oil used for rinsing shall be thrown away and the thief allowed to drain. The thief shall again be inserted into the oil but this time the thumb shall not be removed until the thief reaches the bottom of the container. When the thief is filled, the thumb shall be replaced, the thief quickly withdrawn and the contents allowed to flow into the sample container. The bottom hole shall not be closed with the fingers of the other hand. The free hand shall be used to guide the stream of oil by touching the thief only as necessary. The oil shall not be allowed to flow over the hand or fingers before it flows into the sample container nor shall any excess be allowed to flow over the drum or barrel and back again through the open bunghole. When the sample container is filled it shall be closed quickly and the bung replaced in the drum and tightened. The sample container shall be labeled at once and taken to the testing laboratory as quickly as feasible.

(e) Smaller containers such as cans of 5-gal. capacity or larger shall be sampled with a thief, similar to the drum thief but of proportionately smaller dimensions, using a method similar in general to the one described for drums and barrels in Paragraph (d). To sample a shipment of cans of less capacity than 5-gal. one or more cans shall be selected at random from the shipment using the entire contents of the can (or cans) as the sample.



(f) Tank cars of oil shall be sampled by introducing the tank-car thief through the manhole on top of the car, the cover of which shall be removed carefully so as not to introduce any dirt into the oil. The thief shall be lowered until it strikes the bottom of the tank car when it automatically opens and fills. Under no conditions shall samples be taken from the bottom draw-off.

(g) When separate samples are being taken from a consignment or part of a consignment, care shall be exercised to prevent contaminating the samples. A separate thief shall be used for each sample or the thief previously used shall be well drained and then thoroughly washed with oil from the next container to be sampled, throwing away the oil thus used for washing, before taking the portion reserved for the sample (see Paragraph (d)). Enough thieves shall be provided to insure thorough drainage of each thief after rinsing with oil to be sampled before it is used to withdraw the sample. For obtaining only a few samples, two thieves are enough, but for obtaining a large number of samples (for example, sampling a carload of drummed oil) six or more are desirable.

(h) When one average sample of a consignment or batch is being taken, the same thief may be used throughout the sampling operation, omitting the precaution of rinsing the thief with oil before taking any of the portions that make up the total average sample.

#### Procedure for Sampling Oil in Service

8. When taking samples of oil from transformers, oil circuit breakers and similar electrical apparatus where a thief cannot be used, the precautions outlined in the preceding sections shall be observed so far as practicable. In addition, care shall be taken to procure a sample that represents fairly the oil at the bottom of the tank. Transformer and oil circuit-breaker tanks, and similar

electrical apparatus are usually provided with drain pipes or sampling cocks from which the sample may be allowed to flow into the sample container. Before the sample is taken, enough oil shall first be allowed to flow to waste through these drain pipes or sampling cocks to insure that the sample will not be that portion which was stored in the drain pipe. For this reason, the valve and the drain pipe should be small enough to be emptied with convenience and yet large enough to give an even flow of oil and avoid clogging by sediment. A U. S. Standard  $\frac{1}{4}$ -in. nominal size pipe (equivalent to an inside diameter of approximately 9 mm.) with suitable valve is recommended. This, of course, may be separate from the drainage pipe and valve or may be connected to the drainage valve by means of a suitable reducer.

#### SPECIFIC GRAVITY

##### Procedure

9. Specific gravity shall be determined in accordance with the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.<sup>4</sup>

#### COLOR

##### Procedure

10. Color shall be determined in accordance with the Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155) of the American Society for Testing Materials.<sup>4</sup>

#### VISCOSITY

##### Procedure

11. Viscosity shall be determined in accordance with either the Standard

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.



Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88)<sup>4</sup> or the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445)<sup>4</sup> of the American Society for Testing Materials and unless otherwise specified at a temperature of 37.8 C. (100 F.). The conversion of viscosity values may be made in accordance with the Standard Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (A.S.T.M. Designation: D 446) of the American Society for Testing Materials.<sup>4</sup>

#### FLASH POINT

##### Procedure

12. Flash point shall be determined in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92) of the American Society for Testing Materials.<sup>4</sup>

#### POUR POINT

##### Procedure

13. Pour point shall be determined in accordance with the Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97) of the American Society for Testing Materials.<sup>4</sup>

#### ACID AND BASE NUMBERS

##### Procedure

14. Acid and base numbers shall be determined in accordance with the Tentative Method of Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration (A.S.T.M. Designation: D 663),<sup>4</sup> except that if it is desired to use an electrometric titration method, the Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration (A.S.T.M. Designation: D 664)<sup>4</sup> may be used.

#### SAPONIFICATION NUMBER

##### Procedure

15. Saponification number shall be determined in accordance with the Standard Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 94) of the American Society for Testing Materials.<sup>5</sup>

#### SLUDGE

##### Procedure

16. Sludge formation shall be determined in accordance with the Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil (A.S.T.M. Designation: D 670) of the American Society for Testing Materials.<sup>5</sup>

#### INORGANIC SALTS (CHLORIDES AND SULFATES)<sup>6</sup>

##### Procedure

17. The presence of mineral acids (Cl or SO<sub>3</sub>) shall be determined by thoroughly shaking in a separatory funnel, 100 ml. of the oil with 50 to 100 ml. of hot (90 to 100 C.) distilled water and then separating and drawing off the water layer and testing it qualitatively with the usual procedures for the presence of chlorides or sulfates.

#### FREE AND CORROSIVE SULFUR

##### Apparatus

18. The apparatus shall consist of the following:

(a) *Electrolytic Sheet Copper*.—Approximate dimensions: length, 3 in. (7.62 cm.); width, 0.5 in. (1.27 cm.); thickness, 0.031 in. (0.079 cm.).

(b) *Glass Tube*.—Acid-resistant glass, 125-ml. capacity, fitted with a slotted cork (a test tube of suitable size is satisfactory).

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>6</sup> Editorially revised in July, 1946.

(c) *Bath*.—Suitable bath in which a temperature of from 95 to 110 C. can be maintained.

#### Reagents

19. (a) *Fuming Nitric Acid*, c. p.

(b) *Hydrochloric Acid*, c. p. (sp. gr. 1.19).

(c) *Barium Chloride Solution* (10 per cent).

#### Procedure<sup>7</sup>

20. (a) The copper strip shall be mechanically polished. It shall be thoroughly washed with distilled water to remove the polishing compounds and dried between filter paper. The polished copper strip shall be placed in the glass tube and 100 ml. of oil added. The strip shall be completely immersed. The tube shall be closed with a vented cork and the oil heated in the bath for not less than 5 hr., maintaining the oil sample at a temperature between 95 and 110 C.

(b) If free sulfur or corrosive sulfur compounds are present, the copper strip will be discolored. This discoloration (a dark characteristic film) is not definite proof of the presence of free sulfur or corrosive sulfur compounds, and the film shall be tested as follows:

Remove the oil from the copper strip by rinsing with petroleum ether. After the ether has evaporated, completely dissolve the discoloring film with a few drops of fuming nitric acid, catching the solution in a 50-ml. beaker. In this manner a minimum of copper is removed. Wash the strip with a minimum of distilled water, catching the washings in the beaker. To the combined solution, add about 5 ml. of HCl (sp. gr. 1.19). Boil the solution to drive off free nitric acid. If necessary, dilute with a small amount of distilled water, and then to the boiling solution add 5 ml. of the barium chloride solution (10 per cent). A white precipitate proves

the presence of sulfur. Minute traces of sulfur are not precipitated immediately. If no precipitate forms in 6 hr., the temperature of the solution having been maintained at about 95 C., free sulfur and corrosive sulfur compounds shall be considered absent.

NOTE.—All reagents shall be sulfur-free. A blank should be run with each batch of determinations, following the above procedure, but using water in equal volume instead of oil. With necessary precautions the volume of solution for proving the presence or absence of sulfur should not exceed 20 to 30 ml. throughout the manipulation.

#### STEAM EMULSIFICATION TEST

##### Procedure

21. Steam emulsification shall be determined in accordance with the Standard Method of Test for Steam Emulsion of Lubricating Oils (A.S.T.M. Designation: D 157) of the American Society for Testing Materials.<sup>4</sup>

#### DIELECTRIC STRENGTH

NOTE.—Modification of the test procedure for dielectric strength to make it applicable to oils of high viscosity is under development, see Note, Section 1.

##### Apparatus

22. The apparatus shall be as described in Section 3 of the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>5</sup> The test cup for holding the sample of oil shall be made of a material having a suitable dielectric strength. It shall be insoluble in and unattacked by mineral oil and gasoline and nonabsorbent as far as moisture, mineral oil, and gasoline are concerned. The electrodes in the test cup between which the sample is tested shall be circular disks of polished brass or copper, 1 in. in diameter and having square edges. The electrodes

<sup>7</sup> Editorially revised in January, 1947.

shall be mounted in the test cup having their axes horizontal and coincident, with a gap of 0.1 in. between their adjacent faces, and with top of electrodes about 33 mm. ( $1\frac{1}{4}$  in.) below the top of the cup.

#### Procedure

23. (a) The electrodes and the test cup shall be wiped clean with dry calendered tissue paper or with a clean dry chamois skin and thoroughly rinsed with oil-free dry gasoline until they are entirely free from fibers.

(b) The spacing of electrodes shall be checked with a standard round gage having a diameter of 0.100 in. and the electrodes then locked in position. Care shall be taken not to touch the electrodes with the gage or in any other manner after cleaning the electrodes and cup, so as to avoid any possible contamination.

(c) The test cup shall be filled with dry gasoline, and voltage applied with uniform increase at the rate of approximately 3 kv. per sec. (rms. value) until breakdown occurs. If the dielectric strength is not less than 25 kv., the cup shall be considered in suitable condition for testing the oil. If a lower test value is obtained the cup shall be cleaned with gasoline and the test repeated.

NOTE.—Evaporation of gasoline from the electrodes may chill them sufficiently to cause moisture to condense on their surface. For this reason, after the final rinsing with gasoline, the test cup should be immediately filled with the oil which is being tested and the test proceeded with at once, or the electrodes should be thoroughly dried.

(d) The temperature of the oil when tested shall be the same as that of the room which should be between 20 and 30 C. (68 and 86 F.). Testing at lower temperatures is likely to give variable results which may be misleading.

(e) The sample in the container shall

be shaken so as to thoroughly mix the oil before filling the test cup. This is even more important with used than with new oil as the impurities may settle to the bottom and the test may be misleading.

(f) The cup shall be filled with oil to a height of not less than 20 mm. (0.787 in.) above the top of the electrodes.

(g) The oil shall be gently agitated by rocking the cup and then the oil shall be allowed to stand in the cup for 3 min. before the first puncture, and 1 min. before each succeeding puncture. This will allow air bubbles to escape.

(h) Voltage shall be applied and increased uniformly at a rate of approximately 3 kv. per sec. (rms. value) until breakdown occurs as indicated by a continuous discharge across the gap. (Occasional momentary discharges which do not result in a permanent arc may occur; these should be disregarded.)

(i) Provision shall be made for opening the circuit as promptly as possible after breakdown has occurred in order to prevent unnecessary carbonization of the oil. After each puncture, the testing vessel shall be jarred to loosen particles of carbon adhering to the electrodes and the oil gently agitated but not with sufficient violence to introduce air bubbles.

(j) Five breakdowns shall be made on each filling after which the vessel shall be emptied and refilled with fresh oil from the original sample. The test shall be continued until the averaged values of at least three fillings do not differ from their mean by more than 10 per cent.

(k) The last filling of the cup should drain the container. If a quart sample be received, allowance shall be made in withdrawing portions so that just enough remains for a final filling of the cup with the drainings from the container.

**Report**

24. The report shall include the following:

(1) The volts (rms. value) at each puncture, the average voltage for each of the three or more fillings and the grand average,

(2) The report shall be stated as kv., 1.0-in. disk, 0.1-in. gap, and

(3) The approximate temperature of the oil at the time of the test.

NOTE.—A precision of about 3 per cent may reasonably be expected in 15 tests distributed among three consistent fillings taken in succession. But if the length of the gap is readjusted and possibilities of contamination exist, the precision may be only 6 or 7 per cent. Differences as great as 10 or 12 per cent may occur between different laboratories even where the work is carefully done. (See Appendix I to Report of Committee D-9 of the American Society for Testing Materials in 1921, *Proceedings*, Am. Soc. Testing Mats., Vol. 21, p. 397 (1921).)

# *Standard Method of Test for*

## SAPONIFICATION NUMBER OF PETROLEUM PRODUCTS BY COLOR-INDICATOR TITRATION<sup>1</sup>



A.S.T.M. Designation: D 94 - 45

ADOPTED, 1944; REVISED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 94; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test is intended for determining the amount of constituents in petroleum products that will easily saponify under the conditions of the test. The method is applicable to new or used petroleum oils including electrical insulating oils, and to mixtures of fats and mineral oils. It is not applicable to used internal-combustion-engine crankcase oils and used turbine oils, or to oils containing compounds of sulfur, phosphorus, the halogens, or other elements that consume acids or alkalies.

NOTE 1.—This method does not always give an accurate indication of the saponifiable organic material present because the alkali may be consumed by inorganic or certain organic acids, most nonalkali soaps, free sulfur, sulfurized oils,

etc. The presence of such materials increases the saponification number above that of fatty saponifiable materials for which the method is primarily intended. The odor of hydrogen sulfide near the end of the back-titration in the saponification test is an indication that certain types of reactive sulfur compounds are present in the sample. In the case of other reactive sulfur, chlorine, and phosphorus compounds, and other interfering materials, no simple indication is given during the test. A gravimetric determination of the actual amount of fatty acids is probably the most reliable method for such compounds.

### Definition

2. *Saponification Number.*—The saponification number of an oil is the number of milligrams of potassium hydroxide which is consumed by one gram of oil under the conditions of the test as prescribed in this method.

### Outline of Method

3. The method involves heating a weighed sample of the oil, dissolved in methylethylketone, with a measured quantity of a standard alcoholic solution of potassium hydroxide and determining the amount of unconsumed potassium hydroxide by titration after the heating period, with a standard solution of

<sup>1</sup> Under the standardization procedure of the Society, this method is under the joint jurisdiction of A.S.T.M. Committee D-2 on Petroleum Products and Lubricants and Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1921 to 1928, being revised in 1928. It was adopted in 1928, revised in 1936, but withdrawn and republished as tentative from 1938 to 1944, being revised in 1939, 1941, 1942, 1943, and 1944.

This method comprises a revision and consolidation of the Standard Method of Test for Saponification Number (D 94 - 36) and the Tentative Method of Test for Saponification Number of Electrical Insulating Oils (Modified Baader Method) (D 438 - 36 T), which were discontinued in 1938.

hydrochloric acid. The potassium hydroxide consumed is calculated and divided by the weight of the sample.

#### Apparatus

4. The apparatus shall consist of the following:

(a) *Erlenmeyer Flask and Condenser.*—An Erlenmeyer flask, 300-ml. capacity, alkali-resistant,<sup>3</sup> to which an adequate reflux or mushroom-type condenser is attached by means of a ground-glass joint.

(b) *Hot Plate.*—A suitable hot plate heated by either electricity or steam.

#### Reagents

5. (a) *Alcoholic Potassium Hydroxide (0.5 N).*—Prepare 0.5 N solution by dissolving KOH "purified by alcohol" in the alcohol specified in Paragraph (b). Allow the solution to settle in a dark place. Filter the solution, allow to stand for 24 hr. and standardize to 0.5 N.

(b) *Alcohol.*—Purify 95 per cent ethanol (Note 2), or 95 per cent ethanol to which has been added 10 per cent by volume of methanol (Note 3), with silver oxide in the following manner: Dissolve 1.5 g. of c.p. silver nitrate in about 3 ml. of water, add to 1 liter of alcohol in a glass-stoppered bottle, and mix thoroughly. Dissolve 3 g. of KOH "purified by alcohol" in 10 to 15 ml. of warm alcohol. After cooling, add slowly to the alcoholic silver nitrate solution, stirring slightly. Allow the precipitated silver oxide to settle, siphon off the clear solution, and distill on a steam bath.

NOTE 2.—It has been found that 99 per cent isopropyl alcohol may be substituted for the purified ethanol and entirely satisfactory results obtained. This substitution is not permissible however, in referee tests.

NOTE 3.—This composition is available under the name of: "U. S. Treasury Dept. Specially Denatured Formula 30 (Regulation No. 3—1938)." Formula 3A plus 5 per cent methanol is an equivalent.

<sup>3</sup> Pyrex and Kimble resistant glasses are suitable.

(c) *Standard Hydrochloric Acid Solution (0.5 N).*

(d) *Phenolphthalein Solution.*—Dissolve 1 g. of phenolphthalein in 100 ml. of alcohol (Paragraph (b)).

(e) *Methylethylketone, Technical Grade.*—This should be stored in dark or brown bottles.

#### Blank Determinations

6. Blank determinations shall be made in duplicate on the alcoholic KOH and repeated for each batch of methylethylketone, as follows: Measure accurately from a burette into the Erlenmeyer flask,  $25 \pm 0.03$  ml. of alcoholic KOH and add  $25 \pm 1$  ml. of methylethylketone. Connect the condenser to the flask, and heat for 30 min. (Note 6, Section 8) after refluxing begins. Disconnect the condenser, add 50 ml. of A.S.T.M. precipitation naphtha<sup>4</sup> (Note 7, Section 8), and titrate the blank while hot with 0.5 N HCl, using 3 drops of phenolphthalein indicator. The end point is reached when the indicator color is discharged.

NOTE 4.—The glassware must be chemically clean. It is recommended that flasks be cleaned with chromic acid cleaning solution and rinsed with distilled water.

#### Sample

7. The size of the sample shall be chosen so that the back-titration is from 40 to 80 per cent of the blank, except that the weight of the sample shall not exceed 20 g. (Note 5).

NOTE 5.—The size of sample depends upon the amount of saponifiable matter present. A 4 to 20-g. sample is generally required for blends of mineral oils and fats or fatty oils. If the method is used for fats or fatty oils alone, a 1 to 2-g. sample is generally required for materials having a saponification number higher than 210, and a 2 to 3-g. sample for materials having a saponification number lower than 210.

<sup>4</sup> Requirements for this precipitation naphtha are given in Section 4 of the Standard Method of Test for Precipitation Number of Lubricating Oils (A.S.T.M. Designation: D 91), 1946 Book of A.S.T.M. Standards, Part III-A.

### Procedure

8. Weigh the sample to the nearest 0.01 g., by difference, from a small beaker into the Erlenmeyer flask. Add  $25 \pm 1$  ml. of methylethylketone, followed by  $25 \pm 0.03$  ml. of alcoholic KOH measured accurately from a burette. Connect the condenser to the flask and heat for 30 min. after refluxing begins (Note 6). Immediately disconnect the condenser, cautiously add 50 ml. of A.S.T.M. precipitation naphtha<sup>4</sup> (Note 7), and titrate the solution while hot (without reheating) with 0.5 N HCl, using 3 drops of phenolphthalein indicator. The end point is reached when the indicator color is discharged.

NOTE 6.—For most oils, refluxing for 10 min. is sufficient to complete the saponification. When the sample is known to be free of blown rapeseed or other oils requiring 30-min. refluxing, the time may be shortened to 10 min. In this case the reflux time for the blank determination is also reduced to 10 min.

NOTE 7.—In the case of insulating oils, the addition of A.S.T.M. precipitation naphtha is not necessary.

### Calculation

9. (a) The saponification number shall be calculated as follows:

$$\text{Saponification number} = \frac{DV}{W}$$

where:

D = difference between the number of milliliters of 0.5 N HCl required for the sample in the determination and the average of that required for the two blanks,

V = strength of the 0.5 N HCl expressed as milligrams of KOH per milliliter, and

W = the weight of sample in grams.

(b) The percentage of fatty oil or fat in a compounded petroleum product can be calculated from the saponification number of such a product only when the saponification number of the fatty oil

is known. If the saponification numbers of both fatty oil and compounded oil are known, the following formula may be used:

$$\text{Fatty oil or fat, per cent} = \frac{C}{F} \times 100$$

where:

C = saponification number of the compounded oil, and

F = saponification number of the fatty oil or fat.

NOTE 8.—For this calculation, the following saponification numbers may be used:<sup>a</sup>

Fatty Oil or Fat	Saponification Number
Castor.....	176 to 187
Cottonseed.....	187 to 197
Blown cottonseed.....	210 to 225
Fish.....	140 to 193
Lard oil.....	190 to 198
Neatsfoot.....	193 to 204
Peanut.....	186 to 197
Rapeseed.....	170 to 179
Blown rapeseed.....	195 to 216
Soybean.....	189 to 197
Sperm.....	120 to 140
Tallow.....	193 to 198

<sup>a</sup> Inasmuch as the saponification number of degrades varies between 85 and 150 according to the country of its origin and its degree of refinement, it is not possible to calculate percentages of degrades in compounded products unless the saponification number of the degrades used is known.

### Identification of Fat

10. In an unknown sample, particularly when a low saponification number leaves doubt as to whether fat is actually present, the fatty acid may be extracted (as the soap) and recovered for gravimetric determination. For this purpose, the saponified and neutralized solution (Section 8) shall be treated as in a grease analysis, as described in Section 14 (b) of the Standard Methods of Analysis of Grease (A.S.T.M. Designation: D 128) of the American Society for Testing Materials.<sup>5</sup> The fatty acid is thus obtained and may then be used for subsequent identification tests.

### Reproducibility of Results

11. With proper care and attention to details, determinations by different

<sup>5</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

operators should agree within plus or minus 0.5 saponification numbers for values less than 5.0, and within plus or minus 0.7 saponification numbers for values above 5.0. In the case of straight fats and fatty oils, determinations by different operators should agree

within plus or minus 4.0 saponification numbers.

NOTE 9.—No specific permissible differences can be given for highly colored new or used oils, or for oils which produce dark-colored solutions upon saponification, as color may interfere with the detection of the end point of the titration.



# *Standard Methods of*

## TESTING GLASS SPOOL INSULATORS<sup>1</sup>



A.S.T.M. Designation: D 550 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 550; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods are intended to apply to the testing of glass spool insulators used for secondary power distribution.

### Sampling

2. Sampling for purpose of these test methods shall be as specified. The glass insulators shall be selected in such a manner as to be representative of the lot. Sampling for testing by the purchaser on individual shipments shall be by mutual agreement between the purchaser and the seller.

### Routine Tests

3. Routine tests shall consist of visual inspection for physical and structural defects, gaging for pin cavity formation, and mechanical test for structural strength, applied in that order. Mechanical strength tests shall be made only on insulators which are free from objectionable physical and structural defects and have passed visual inspection.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1939 to 1944, being revised in 1941.

### Structural Defects

4. The finish of the insulators as indicated by the presence or absence of structural defects, such as cracks, chips, folds, and stones, or other visible flaws covered in the purchaser's product specifications shall be determined by visual inspection.

Chips are defects caused by the fracture and complete removal of a portion of the insulator.

Folds are linear indentations of the surface of the insulator caused by the meeting and partial welding of a re-entrant portion of a surface which has been partially chilled in some previous stage of the process of manufacture.

Stones are foreign, solid, inorganic matter completely or partially surrounded by glass, usually consisting of particles of refractory material, crystalline products due to combination of refractories and glass batch constituents, particles of an incompletely melted batch, or products of devitrification of the glass.

### Gaging

5. The gaging test is intended to determine whether the pin cavity is

properly formed with smooth and uniform bore. The gage shown in Fig. 1 shall be inserted in the pin cavity of the glass insulator and observation shall be made of the fit.

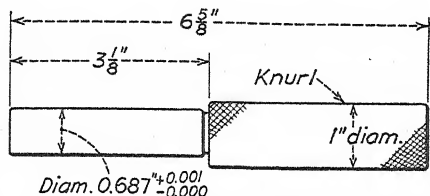


FIG. 1. Gage for Testing Pin Cavity.

### Mechanical Strength

6. The insulator shall be mounted in a supporting structure (Fig. 2), ap-

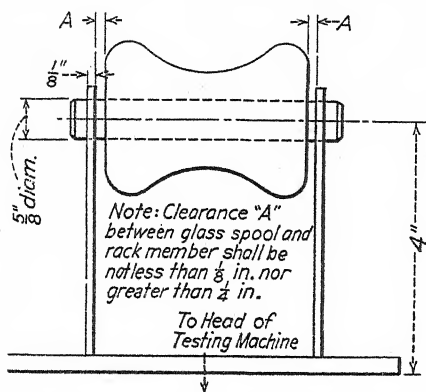


FIG. 2.—Assembly for Mechanical Strength Test.

proximating a section of a standard secondary rack, which shall be fastened to one head of the tension testing machine. The load shall be applied mechanically at right angles to the rod through the glass insulator by means of a  $\frac{3}{8}$ -in. flexible 6 by 19 wire rope in the wire groove of the insulator. The method of applying the load is shown in Fig. 3 ( $\frac{1}{16}$  copper-weld shackles procurable from line hardware manufacturers may be substituted). The load shall be increased from 75 per cent of the rated strength of the insulator to failure at a

rate per minute of not less than 30 nor more than 60 per cent of the rated strength of the insulator. The strength of the insulator shall be considered as the load at the first sign of failure.

### Report

7. A report shall be made of tests made on each day's production of glass insulators and shall include the following:

(1) A description of the material, the name and type, date of manufacture,

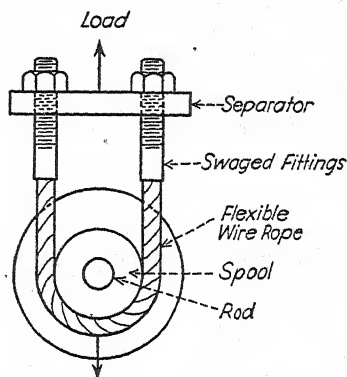


FIG. 3.—Shackle for Mechanical Strength Test of Glass Spool Insulators.

and number of insulators in the day's production.

(2) The number of insulators tested and the results of each test, including the minimum and average value of the mechanical strength tests in pounds.

### Record

8. The results obtained shall be preserved as a record.

### Marking

9. Glass insulators inspected and tested during production in accordance with these methods of test may be so designated by imprinting on the container the date of manufacture, and the statement that they were tested in accordance with A.S.T.M. Methods D 550-44, together with a reference to the test report by number.

# *Standard Methods of*

## TESTING PIN-TYPE, LIME-GLASS INSULATORS<sup>1</sup>



**A.S.T.M. Designation: D 468 - 46**

ADOPTED, 1942; REVISED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 468; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods are intended to apply to the testing of two types of pin-type lime-glass insulators:

(1) Those having a dry flashover of 85,000 v. or less, and used for supporting conductors of primary and secondary power distribution systems.

(2) Those used for supporting conductors of open-wire communication and signal lines.

### ROUTINE TESTS

2. Routine tests shall consist of visual inspection for physical and structural defects; electrical characteristics of insulating material as to dielectric constant and dissipation factor; thermal shock test for homogeneity and uniformity of annealing; gaging for pin-hole formation; and mechanical test for structural strength, the tests being applied in that order. Thermal shock and mechanical strength tests shall be made only on insulators which are free from objectionable physical and structural defects and have passed visual inspection. Separate insulators shall be used for each

test, and insulators which have been gaged or tested shall be discarded.

### Structural Defects

3. The finish of the insulators as indicated by the presence or absence of structural defects, such as cracks, chips, folds, and stones, or other visible flaws covered in the purchaser's product specifications shall be determined by visual inspection.

Chips are defects caused by the fracture and complete removal of a portion of the insulator.

Folds are linear indentations of the surface of the insulator caused by the meeting and partial welding of a re-entrant portion of a surface which has been partially chilled in some previous stage of the process of manufacture.

Stones are foreign, solid, inorganic matter completely or partially surrounded by glass usually consisting of particles of refractory material, crystalline products due to combination of refractories and glass batch constituents, particles of an incompletely melted batch, or products of devitrification of the glass.

### Electrical Characteristics

4. Dielectric constant and dissipation factor shall be measured on plaque

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1937 to 1942, being revised in 1938, 1939, and 1942.

specimens of the same material as that used in producing the insulators. The size of specimens and the measurement method shall be in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

### Thermal Shock Test

5. (a) The thermal shock test consists in transferring the insulators in a horizontal position from a hot-water bath to a cold-water bath and then from a cold to a hot bath. The transfer may be performed manually or with the aid of a machine designed for the purpose. In the latter case certain precautions in handling are desirable as outlined below. The methods are believed to be equivalent<sup>4</sup> and are intended to be used interchangeably; the maintenance of temperature and the agitation of the baths compensate for the slightly longer time of transfer in the machine method. When these methods are made the basis of a purchase specification the purchaser's choice of method shall prevail or control.

(b) *Water Baths*.—Two water baths are required. The containers may be made of planking. If metal containers are employed, they shall be insulated with 1 in. of cork or equivalent material to prevent excessive cooling at the sides. Provision shall be made for thoroughly stirring the water in both baths in the manual method, and in this method the volume of water in each bath shall be at least 50 times the volume of the glass immersed. In the machine method both baths shall be equipped with means for keeping the water agitated so that it circulates thoroughly about the bath at all times during the test period. The

temperature of both baths shall be maintained within plus or minus 2 F. of the specified value throughout the immersion period, and the volume of water in each bath shall be at least 20 times the volume of glass immersed.

One bath shall be equipped with means for heating the water and the heating device shall be so placed that the bath will be heated uniformly when the normal agitation is employed. The bath should not be heated by any method which applies heat directly to the walls of the container such as heating the container on a hot plate. In the manual method no heat is to be added during the period of immersion; the hot water may be poured into the container for each test if a supply of preheated water is used. In the machine method heat is added as may be necessary to maintain the temperature.

The other bath shall be equipped with means for introducing cold water as required. If ice is used to adjust the temperature of the cold bath, it shall be removed from the bath and the water stirred before immersing the insulators.

(c) *Rack*.—A rack for immersing the insulators in the baths shall be so arranged as to keep the insulators separated from each other and to support them in a substantially horizontal position. There should be holes in the base of the rack and in the sides between the slats so as to permit free circulation of the water of the baths between the insulators when immersed and so as to permit a uniformly smooth transfer of the insulators without causing excessive agitation at the time of immersion.<sup>5</sup> There shall be a clearance of at least 2 in. between the outermost insulators and the walls of the bath.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> For a comparison of results obtained by the two procedures see K. C. Lyon and R. F. Scott, "Thermal Shock Testing of Lime-Glass Insulators," A.S.T.M. Standards on Electrical Insulating Materials, p. 469 (1944).

<sup>5</sup> An automatic mechanical apparatus suitable for this test may be adapted from that shown in Fig. 1 of the Standard Method of Thermal Shock Test of Glass Containers (A.S.T.M. Designation: C 149), see 1946 Book of A.S.T.M. Standards, Part II, and for which detail drawings are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.



amed for presence or absence of cracking, chipping, and spalling.

### Gaging Threads

6. The gaging test is intended to determine whether the pin hole is properly formed with smooth and uniformly

gaged and when so seated, observation shall be made of the clearance in inches between the top end of the gage and the crown of the insulator. There shall be no perceptible play or rocking of the insulator when seated on the gage. The number of turns necessary to release

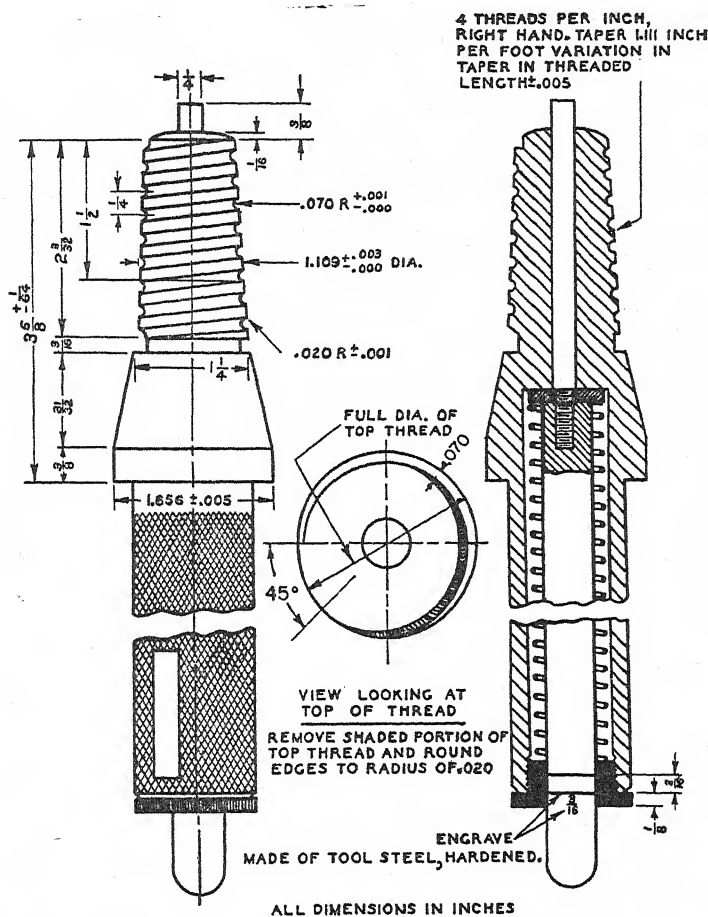


FIG. 2.—Gage for Wooden-Pin Insulators.

pitched threads. Separate gages are required for the several types of insulators. Figure 1 shows the gage for power insulators, Fig. 2 is the gage for wooden-pin communication insulators, and Fig. 3 gives the gage for communication insulators (type CSC). The insulator shall be screwed home on the appropriate

the gage shall be determined by holding the insulator upside down and counting the revolutions of the insulator required before it can be disengaged from the gage.

### Mechanical Strength Test

7. The insulator to be tested shall be

mounted on the steel pin detailed in Fig. 4, provided with a threaded thimble or cap of lead containing not over 5 per cent of antimony, so mounted as to furnish sufficient strength to prevent permanent deflection during the pull

up in a practically stepless variation to the failure point. The load shall be increased rapidly to approximately 75 per cent of the rated strength of the insulator and at a slower rate from this point to failure. The rate of increase

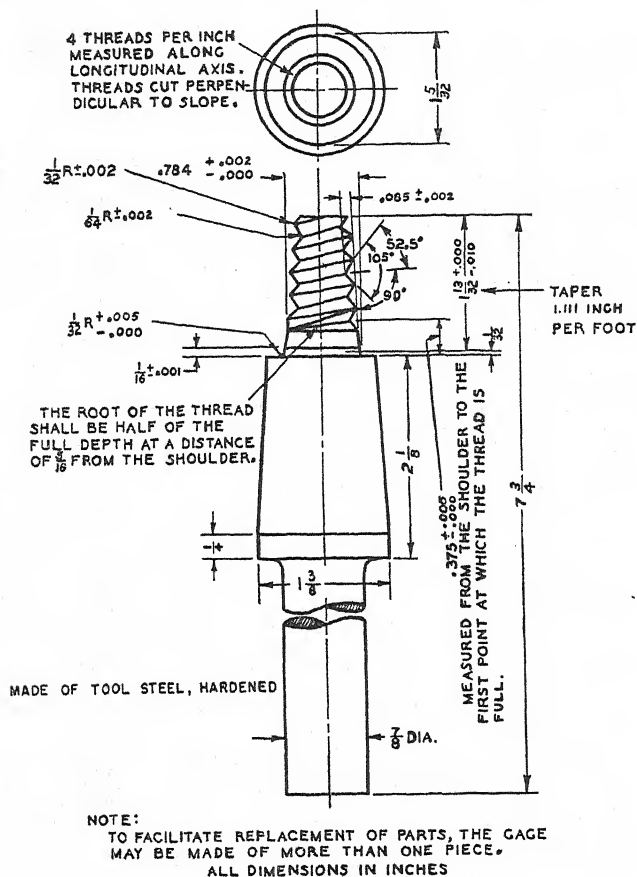


FIG 3.—Gage for Carrier-Current Insulators (CSC).

required to fail the insulator. The load shall be applied mechanically at right angles to the pin by means of a  $\frac{3}{8}$ -in. flexible 6- by 19-wire rope in the side groove of the insulator. The method of applying the load is shown in Fig. 5 ( $\frac{7}{16}$  copper weld shackles procurable from line hardware manufacturers may be substituted). The load shall be started at zero and smoothly brought

up in a practically stepless variation to the failure point. The load shall be increased rapidly to approximately 75 per cent of the rated strength of the insulator and at a slower rate from this point to failure. The rate of increase

Class of Insulator	Type of Test	Increase in Load per Minute in Percentage of Rated Strength	
		Min.	Max.
Pin-type	Cantilever	30	60

The strength of the insulator shall be the total load at first visible crack or fracture of the glass.





electrodes than one and one-half times the specimen dry arcing distance, with a minimum allowable spacing of 3 ft.

(d) *Voltage Application*.—The initial applied voltage may be quickly raised to approximately 75 per cent of the expected average dry-flashover value. The continued rate of voltage increase shall be such that the time to flashover will be not less than 5 sec. nor more than 30 sec. after 75 per cent of the flash-over value is reached.

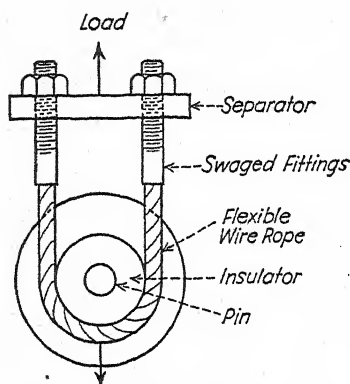


FIG. 5.—Shackle for Mechanical Strength Test of Glass Pin-Type Insulators.

(e) *Dry-Flashover Voltage Value*.—The dry-flashover voltage value of a specimen shall be the arithmetic mean expressed in rms. volts of not less than five individual flashovers taken consecutively. The period between consecutive flashovers shall be not less than 15 sec. nor more than 5 min.

### Low-Frequency Dry-Flashover Corrections

9. (a) *Standard Conditions*.—Dry-flashover values shall be corrected to the following standard conditions:

Barometer.....29.92 in. (760 mm.) of mercury  
 Temperature.....77 F. (25 C.)  
 Vapor pressure.....0.6085 in. (15.45 mm.)

(b) *Air Density*.—The dry-flashover value shall be corrected to standard air density in accordance with the American Standard for Measurement of Test Voltage in Dielectric Tests (ASA No.: C68.1-1942). The relative air density (RAD) shall be calculated as follows:

$$\text{RAD} = \frac{17.9B(\text{in.})}{459 + t(\text{F.})}$$

or

$$\text{RAD} = \frac{0.392B(\text{mm.})}{273 + t(\text{C.})}$$

where:

$t$  = room temperature, and  
 $B$  = barometric pressure

(c) *Humidity*.—The low-frequency dry-flashover value shall be corrected to standard humidity conditions in accordance with the tentative curve shown in Fig. 6.

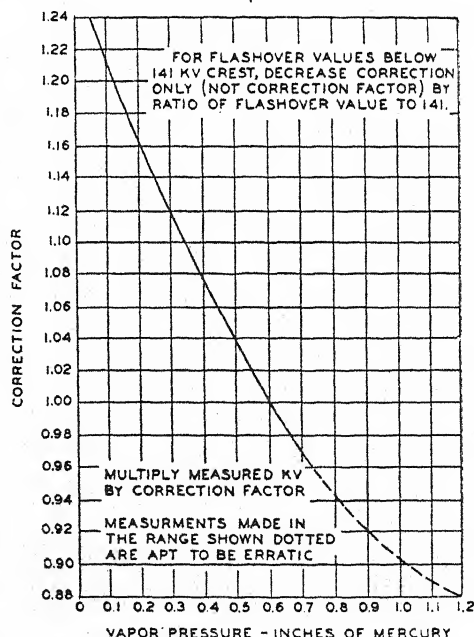


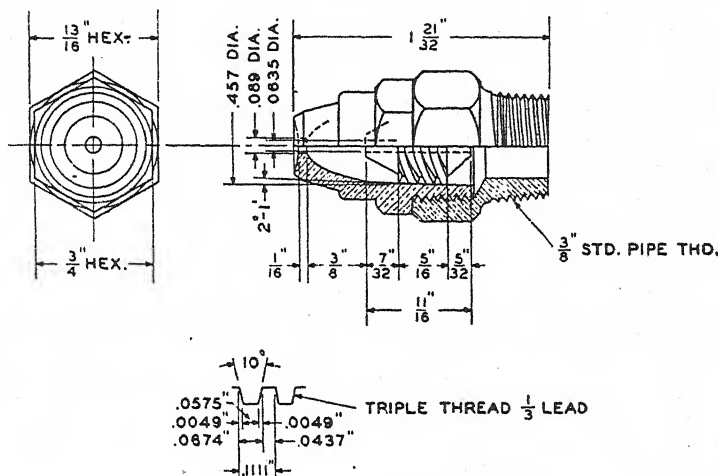
FIG. 6.—Low Frequency Humidity Correction Factors.

The vapor pressure shall be determined by the following procedure: Humidity shall be measured with wet and dry bulb thermometers, the air being circulated past the thermometers at a velocity of 3 m. (9.84 ft.) or more per second or by the sling psychrometer. The measurements shall be reduced to vapor pressure with the assistance of the Smithsonian

$t'$  = temperature of the wet bulb thermometer in degrees Fahrenheit or Centigrade, and

$B$  = barometric pressure in inches or millimeters.

(d) *Reproducibility*.—Due to differences in atmospheric and other uncontrollable conditions, even with properly standardized equipment, a vari-



DETAILS OF THREADS ON INTERNAL ELEMENT

FIG. 7.—Spray Nozzle.

Meteorological Tables or by the following formula:

*English Units:*

$$E = E' - 0.000367 B (t - t') \left( 1 + \frac{t' - 32}{1571} \right)$$

*Metric Units:*

$$E = E' - 0.000660 B (t - t') (1 + 0.00115 t')$$

where:

$E$  = vapor pressure in inches or millimeters,

$E'$  = pressure of saturated aqueous vapor at temperature  $t'$ ,

$t$  = temperature of air in degrees Fahrenheit or Centigrade,

ation of plus or minus 5 per cent from the probable true average dry-flashover value may be expected in tests conducted in one laboratory, and plus or minus 8 per cent in values obtained in tests conducted in different laboratories.

#### Low-Frequency Wet-Flashover Test

10. (a) *Mounting Arrangement*.—The test specimen shall be mounted in accordance with Section 8 (a), (b) and (c).

(b) *Spray Nozzles*.—The insulator being tested shall be placed in an area supplied with a reasonably uniform artificial precipitation produced by a suitable number of spray nozzles of the design shown in Fig. 7 or equivalent.

(c) *Direction of Spray*.—The spray shall be so directed as to strike the insulator downward at an angle of 45 deg. from the vertical and parallel to the vertical plane through its axis within practical limitations.

(d) *Rate of Precipitation*.—The standard rate of precipitation at the specimen shall be 0.2 in. (5.08 mm.) per min. when measured as described in Paragraphs (i), (j), (k), (l), (m).

(e) *Resistivity of Spray Water*.—The standard resistivity of the spray water shall be 7000 ohms per cu. in. (17,800 ohms per cu. cm.) at the temperature at which the water strikes the specimen. The resistivity of the spray water shall not deviate more than 15 per cent from the standard.

(f) *Temperature of Spray Water*.—The temperature of the spray water when it strikes the specimen shall not deviate more than 15 C. from room temperature.

NOTE.—No exact data are available on the effect of variation in water temperature. Experience indicates, however, that normal variations in water temperature are unimportant provided the correct resistivity is maintained.

(g) *Pressure of Spray Water*.—The spray water shall be delivered to the nozzles at pressure of not less than 35 psi. (2.46 kg. per sq. cm.) and at not more than 60 psi. (4.22 kg. per sq. cm.) measured near the nozzle.

(h) *Area Sprayed*.—The specimen shall be centrally located by inspection in an area uniformly sprayed having a width at least two times and a length at least one and one-fourth times that of the specimen being tested.

(i) *Location of Spray Equipment*.—Spray nozzles, supply pipes, and the supporting structure for them shall be located not closer than 3 ft. from the specimen.

(j) *Measuring Vessel*.—The vessel used to collect the water for measuring the precipitation, resistivity, and tem-

perature shall have a top opening 6 to 12 in. in inside diameter with an up-standing rim at least 1 in. in height having an edge thickness not exceeding  $\frac{1}{16}$  in.

(k) *Position of Measuring Vessel*.—During measurements of precipitation, the measuring vessel shall be held opposite the top of the insulating unit of the specimen in line between the spray nozzle and the axis of the specimen and at a point approximately 3 in. outside the largest diameter of the insulating element. The vessel shall be held with its top opening horizontal.

(l) *Duration of Individual Measurements*.—Final measurements of the rate of precipitation shall be accurately timed and shall be of a duration of at least 1 min. for each measurement.

(m) *Tolerances on Rate of Precipitation*.—For precipitation measured in accordance with Paragraph (k) and (l), the measurement shall not deviate more than 10 per cent from the standard.

(n) *Constancy of Spray Conditions*.—Care shall be exercised to see that the nozzle pressure and other spray conditions remain practically constant while the tests are being made after final adjustments of the precipitation are completed.

(o) *Preliminary Wetting*.—At not more than 1 min. prior to placing the insulator in the spray, the entire surface of the insulator shall be given a thorough preliminary wetting either by immersion, by spraying in an inverted position, or by using a hose on the insulator in its test position. Water used for preliminary wetting shall have the same resistivity as the spray water.

(p) *Voltage Application*.—At not less than 1 min. after the final adjustment of the spray the applied voltage may be raised quickly to approximately 75 per cent of the expected average wet-flash-over value. The continued rate of

voltage increase shall be such that the time to flashover will not be less than 5 sec. nor more than 30 sec. after 75 per cent of the flashover value is reached.

(q) *Wet-Flashover Voltage Value*.—The wet-flashover voltage value of a specimen shall be the arithmetic mean expressed in rms. volts of not less than five individual flashovers taken consecutively. The period between consecutive flashovers shall be not less than 15 sec. nor more than 5 min.

#### Low-Frequency Wet-Flashover Corrections

11. (a) *Standard Conditions*.—Wet-flashover values shall be corrected in accordance with Section 9 (a) and (b).

(b) *Humidity*.—No correction for humidity shall be made on wet-flashover values.

(c) *Reproducibility*.—Due to variations in water spray and differences in atmospheric or other uncontrollable conditions, even with properly standardized equipment, a variation of plus or minus 8 per cent from the probable true average wet-flashover value may be expected in tests conducted in one laboratory and plus or minus 12 per cent in values obtained in tests conducted in different laboratories.

#### Impulse-Flashover Test

12. (a) *Mounting Arrangement*.—The test specimen shall be mounted in accordance with Section 8 (a), (b), and (c).

(b) *Impulse-Voltage Wave*.—All tests shall be made with a  $1\frac{1}{2}$  by 40 microsecond wave.

(c) *Determination of Impulse-Flashover Voltage*.—The critical impulse-flashover voltage may be determined by either of the following methods:

METHOD No. 1.—The critical impulse-flashover voltage shall be determined by applying impulses of varying magnitude to the specimen and measuring the crest

voltage of the impulse which will cause flashover on approximately 50 per cent of the applications. Impulses shall be applied in increasing or decreasing voltage steps, such that at the highest step flashover occurs on every impulse and at the lowest step no flashover occurs. At least three impulses shall be applied at each voltage step and the voltage differential from one step to the next shall not exceed 5 per cent of the expected critical impulse-flashover voltage. In cases where the voltage spread from the highest to the lowest step is relatively great, the critical impulse-flashover voltage may be determined by plotting an average curve of crest voltage *versus* flashover frequency. The crest voltage at the point where the flashover frequency is 50 per cent shall be taken as the critical impulse flashover.

METHOD No. 2.—The critical impulse-flashover voltage shall be obtained by applying not less than eight impulses of varying magnitude to the specimen so that flashover occurs on not less than 30 and not more than 70 per cent of the total impulses applied. The critical impulse-flashover voltage shall be the crest value of the lowest impulse causing flashover. At least one full impulse wave shall be greater than 95 per cent of the critical impulse-flashover voltage.

(d) *Volt-Time Flashover Curves*.—The complete impulse-flashover characteristics of a specimen tested with the  $1\frac{1}{2}$  by 40 microsecond wave may be presented by means of volt-time curves. Data for plotting volt-time curves shall be obtained by increasing the excitation voltage of the impulse generator in percentage steps of the excitation voltage at the approximate critical flashover. The voltage may be measured by means of a parallel-sphere gap or a cathode-ray oscillograph and the time to flashover may be measured by means of a cathode-ray oscillograph.

Enough test points shall be determined to define the curve within the required limits. Volt-time curves shall be plotted as a smooth line representing the most probable mean of the data points and terminating at the longest and shortest measured times.

### Impulse Flashover Corrections

13. (a) *Standard Conditions*.—The critical impulse-flashover values shall be corrected in accordance with Section 9 (a) and (b).

(b) *Humidity*.—The critical impulse-flashover value shall be corrected to standard humidity conditions in accordance with the tentative curves shown in Fig. 8.

The vapor pressure shall be determined in accordance with the procedure described in Section 9 (c).

(c) *Volt-Time Curves*.—The full humidity correction shown on the curves in Fig. 8 shall be applied to the critical flashover value. For volt-time curves the correction shall be made as follows:

(1) When the critical flashover value occurs at more than 10 microseconds, full corrections shall be applied to all values with time lags of 10 microseconds or more. When flashover occurs at less than 10 microseconds, the correction shall be reduced in the direct ratio that the time to flashover bears to 10 microseconds.

(2) When the critical flashover value occurs at less than 10 microseconds, the correction shall be reduced in the direct ratio that the time to flashover bears to the time at the critical flashover.

(d) *Reproducibility*.—Due to differences in atmospheric and other uncontrollable conditions, even with properly standardized equipment, a variation of plus or minus 5 per cent from the probable true average critical impulse-flashover value may be expected in tests conducted in one laboratory and plus or

minus 8 per cent in values obtained in tests conducted in different laboratories. In volt-time curves similar variations are to be expected at points near the critical impulse-flashover value with considerably larger variations involved as the time to breakdown decreases.

### REPORT

#### Report

14. A report shall be made of tests made of each day's production of insulators and shall include the following:

(a) A description of the material, the name and type, date of manufacture,

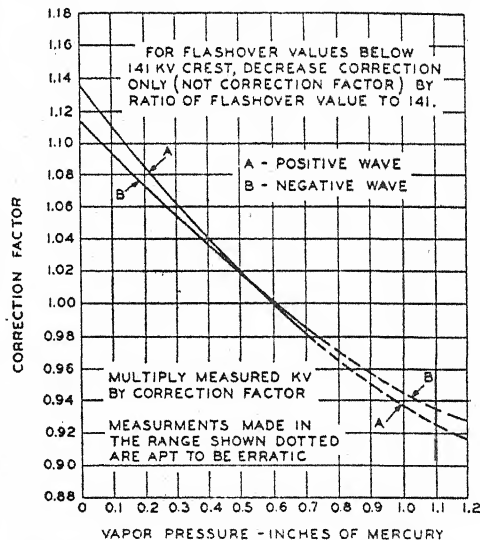


FIG. 8.—Impulse Humidity Correction Factors.

and number of insulators in the day's production.

(b) The number of insulators tested and the results of each of the routine tests, including:

(1) The hot to cold and cold to hot thermal shock sustained,

(2) The minimum number of turns to release the gage, and limits of clearance in inches between the top end of the gage and the crown of the insulator,

(3) The minimum and average value of the mechanical strength tests in pounds.

**Record**

15. The results obtained shall be preserved as a record.

**Marking**

16. Insulators inspected and tested

during production in accordance with these methods of test may be so designated by imprinting on the container the date of manufacture, and the statement that they were tested in accordance with A.S.T.M. Methods D 468, together with a reference to the test report by number.

# *Standard Methods of*

## TESTING ELECTRICAL PORCELAIN<sup>1</sup>



A.S.T.M. Designation: D 116 - 44

ADOPTED, 1930; REVISED, 1934, 1939, 1942, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 116; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods outline procedures for making the various tests described when they are required for an investigation or examination of porcelain or similar vitrified ceramic materials used as electrical insulating mediums. They are not to be interpreted as forming test specifications for complete insulators.

NOTE.—The glaze on a porcelain object may affect certain properties of the material, depending not only upon its presence but also upon its thickness and composition. While the test methods have been written with the intent of applying to unglazed specimens, they are equally suitable for glazed specimens. The report of test should in all cases indicate whether the specimens are glazed or unglazed.

### TENSILE STRENGTH

#### Apparatus

2. Any universal testing machine of suitable capacity may be used provided the error in the loading range does not exceed 1 per cent.

### Test Specimens

3. (a) The tension test specimens shall be of the form and dimensions shown in Fig. 1.

(b) For wet-process porcelain, the test specimens shall be made from a column obtained by forcing the batch mixture through a die. Sections of the column, after thorough drying, shall be formed to the required shape with due allowance for shrinkage. During the firing, care shall be taken to maintain the axis perfectly straight.

(c) For cast porcelain, the specimens shall be cast in molds of calcined gypsum (plaster of Paris) having the required shape, due allowance being made for all shrinkages. None of the material except the fins shall be removed from the specimen.

(d) For dry-process porcelain, the specimens shall be formed by pressure into the required shape in metal molds with due allowance for all shrinkages.

### Mounting of Specimens

4. (a) Suitable grips for holding the specimen shall be self-aligning and shall grip the specimen over sufficient area

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1921 to 1930, being revised in 1924, 1926, 1927, 1928, and 1929.

and in a manner not to cause pinching near the minimum section nor local compression at the smaller ends of the arranged like gimbals or a universal joint used for maintaining a mariner's compass in position. A yoke allows a

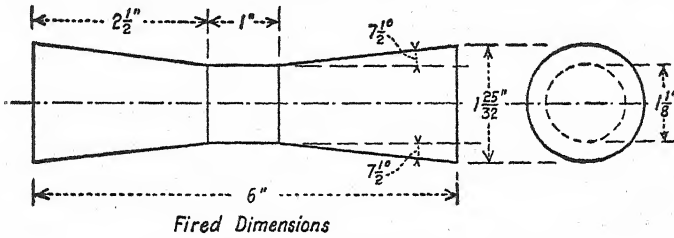


FIG. 1.—Specimen for Tensile Strength Test.

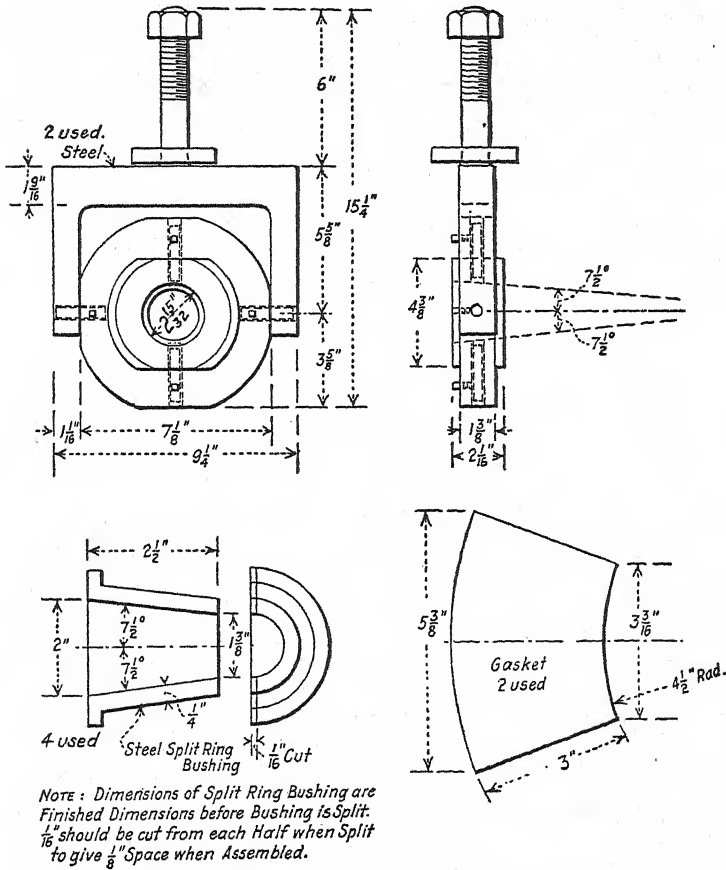


FIG. 2.—Grips for Tension Testing of Porcelain.

metal grips. Grips designed for the purpose and found satisfactory are shown in Fig. 2. In principle they are

ring to swing about a horizontal axis. A smaller ring is situated inside the first one but pivoted by means of pins in a



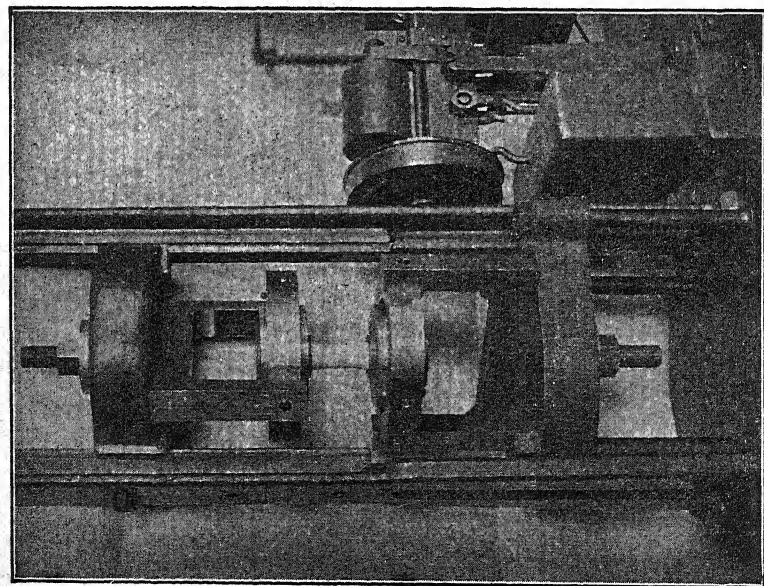


FIG. 3.—Grips and Test Specimens Assembled in Testing Machine in Tension Testing of Porcelain.

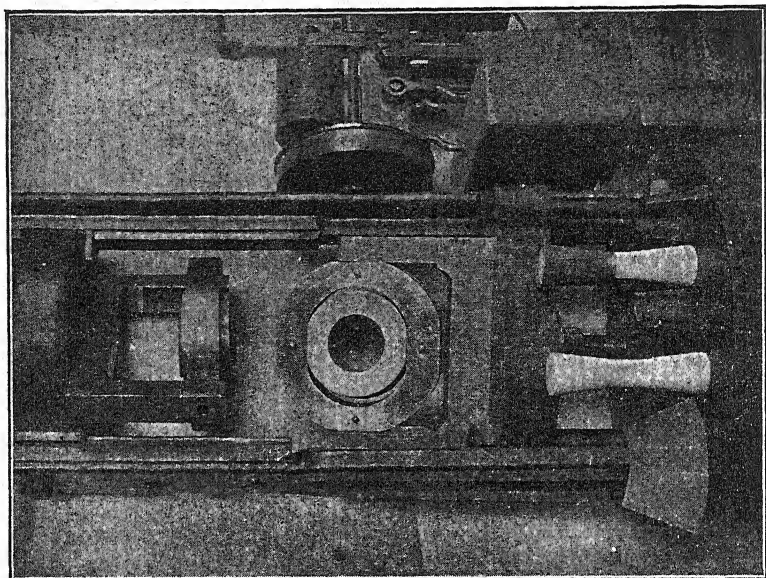


FIG. 4.—Apparatus for Tension Testing of Porcelain Including Grips, Specimen, Split Ring and Blotting Paper Gasket.

plane perpendicular to the horizontal axis. The inner ring is conically shaped and holds the porcelain specimen in place with the aid of the split bushings. The two yokes are placed in the testing machine at right angles to each other.

(b) The test specimen shall be fitted with a compressible gasket around each conical end with the parallel edges nearly touching. Blotting paper or sheet lead  $\frac{3}{8}$  in. in thickness has been found satisfactory. The gasket may be held in place, while assembling the test specimen in the grips, by a temporary clamp at the place of minimum section. After the specimen has been arranged in place ready for test, the temporary clamp shall be removed. The grips holding the test specimen in place in the testing machine are shown in Fig. 3. A disassembled view of the apparatus is shown in Fig. 4.

### Procedure

5. (a) The diameter of each specimen at the minimum section shall be measured with a micrometer graduated to 0.001 in. (0.025 mm.). The value of the diameter to be used in the calculation shall be the average of two measurements taken at right angles.

(b) The tests shall be made at a temperature of about 20 C. (68 F.).

(c) The speed of head of the testing machine shall be such that the load can be accurately weighed.

(d) Not less than five specimens shall be tested.

(e) Results on specimens which do not break in the section of minimum cross-section shall be included, although the unit stress shall be calculated using the minimum cross-section. Any results whose deviation from the mean of the deviations is more than three times the mean of the deviations shall be discarded.

### Report

6. The report shall include the following:

(1) The breaking load of each specimen in pounds,

(2) The diameter of each specimen in inches at the place of break and at the minimum section,

(3) The tensile strength of each specimen in pounds per square inch,

(4) The average of the results given in Item (3) and the percentage average deviation from the mean,

(5) A description of each fracture and the behavior of the material under load. The description of the fracture should indicate whether the fractured area was totally or partially rough, and whether pin holes or other flaws were found.

NOTE.—An examination of the fracture of a normal specimen will show the entire area to consist of a very coarse granular surface, indicating that the entire area was subjected to tension and that the entire surface resisted it. In a specimen containing a pin hole or other flaw, the fracture will usually start near that flaw or pass through it. Part of the surface further away from the flaw will be granular, but the surface near the flaw will be much smoother. If the specimen is compressed at a narrow portion near one of the grips, the surface is again divided into two distinct areas, the area of tension and the area of shear, the latter radiating from the spot which was most under compression.

(6) The rate of application of the load,

(7) The name and rating of the machine used to make the tests, and

(8) A curve sheet showing the tensile strength in pounds per square inch of each specimen, the first value being the lowest and each succeeding one the next higher.

### COMPRESSIVE (CRUSHING) STRENGTH

#### Apparatus

7. Any universal testing machine of suitable capacity may be used provided

the error in the loading range does not exceed 1 per cent.

### Test Specimens

8. The compression test specimens shall be in the form of cylinders and shall be 1.125 in. (2.86 cm.) in length and 1.125 in. (2.86 cm.) in diameter. They shall be made as described in Section 3 (b), (c), and (d). The flat surfaces shall be made as smooth and parallel as possible before firing. After firing, the flat surfaces shall be ground smooth and parallel.

### Procedure

9. (a) A contact pad or cushion of blotting paper  $\frac{1}{8}$  in. in thickness shall be placed between the upper and lower faces of the specimen and the self-aligning heads of the testing machine to equalize irregularities in the surfaces. Fresh cushions shall be used for each specimen.

(b) The diameter and length of each specimen shall be measured with a micrometer graduated to 0.001 in. (0.025 mm.). The value of the diameter to be used in the calculations shall be the average of two measurements taken at right angles.

(c) The tests shall be made at a temperature of about 20 C. (68 F.).

(d) The speed of head of the testing machine shall be such that the load can be accurately weighed.

(e) Not less than five specimens shall be tested.

### Initial and Ultimate Failure

10. Note shall be made of the load in pounds at initial failure, but the value of the load in pounds at ultimate failure shall be determined and used in calculating the compressive (crushing) strength.

### Report

11. The report shall include the following:

(1) The load in pounds on each specimen at initial failure and the load in pounds at ultimate failure,

(2) The diameter and length of each specimen in inches,

(3) The compressive (crushing) strength of each specimen in pounds per square inch,

(4) The average of the results given in Item (3) and the percentage average deviation from the mean,

(5) The behavior of the material under load,

(6) The rate of application of the load,

(7) The name and rating of the machine used to make the tests, and

(8) A curve sheet showing the compressive strength in pounds per square inch of each specimen, the first value being the lowest and each succeeding one the next higher.

### FLEXURAL STRENGTH

#### Apparatus

12. Any universal testing machine of suitable capacity may be used.

#### Test Specimens

13. The flexure test specimens shall be in the form of cylinders and shall be 6 in. (15.2 cm.) in length and 1.125 in. (2.86 cm.) in diameter. They shall be made as described in Section 3 (b), (c) and (d).

#### Mounting of Specimens

14. The specimen shall be supported on two steel wedge-shaped blocks with supporting edge rounded to 0.125-in. (3.175-mm.) radius. The supports shall be 5 in. (127 mm.) apart and the specimen shall be laid upon them with  $\frac{1}{2}$  in. (12.7 mm.) overlap at each end.

The load shall be applied on top of the specimen at right angles to the specimen and midway between the supports by means of a wedge-shaped pressure piece (angle 45 deg.), the edge of which is rounded on a 0.125-in. (3.175-mm.) radius.

### Procedure

15. (a) The diameter of each specimen shall be measured with a micrometer graduated to 0.001 in. (0.025 mm.). The value of the diameter to be used in the calculations shall be the average of two measurements taken at right angles at the middle of the specimen.

(b) The tests shall be made at a temperature of about 20 C. (68 F.).

(c) The load shall be applied at that constant rate of speed which will permit the beam to be kept well balanced at all loads.

(d) Not less than five specimens shall be tested.

### Calculations

16. The modulus of rupture for each specimen shall be calculated as follows:

$$M = \frac{8 Pl}{\pi d^3}$$

where:

$M$  = modulus of rupture,

$P$  = load in pounds at rupture,

$l$  = distance between supports in inches,

$d$  = diameter of specimen in inches, and

$\pi = 3.1416$ .

### Report

17. The report shall include the following:

(1) The breaking load of each specimen in pounds,

(2) The diameter and length of each specimen in inches,

(3) The modulus of rupture for each specimen as calculated in Section 16,

(4) The average of the results given in Item (1) and the percentage average deviation from the mean,

(5) A description of each fracture and the behavior of the material under load,

(6) The rate of application of the load,

(7) The name and rating of the machine used to make the tests, and

(8) A curve sheet showing the flexural strength (modulus of rupture) for each specimen, the first value being the lowest and each succeeding one the next higher.

## RESISTANCE TO IMPACT

### Procedure

18. Resistance to impact shall be determined in accordance with the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256) of the American Society for Testing Materials,<sup>3</sup> using the "Simple Beam Method."

## DIELECTRIC STRENGTH

### Apparatus

19. The apparatus shall conform to the requirements prescribed in Section 3 of the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>3</sup>

### Test Specimen

20. The dielectric strength test specimen shall be a disk approximately 2 in. in diameter, the flat sides being made plane and parallel. The thickness of the specimen shall be 0.250 in. (6.35 mm.), 0.4 in. (10.16 mm.), 0.75 in. (19.05 mm.), or 1.0 in. (25.4 mm.). The

<sup>3</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

thickness shall be within plus or minus 10 per cent of these values.

NOTE.—The specimen tested should be of a thickness comparable to that of the finished porcelain product.

#### Procedure

21. (a) The specimen shall be tested between metallic electrodes 0.75 in.

In applying the sealing wax, one end of the tube is dipped about  $\frac{3}{8}$  in. (9.5 mm.) into a bath of molten wax. Upon removal, it is immediately pressed into place on the specimen and allowed to cool to room temperature. (If a film of wax covers the tube it should be broken before placing the tube on the specimen.) In order more thoroughly to smooth out the wax and seal the tube, the wax seal may be softened slightly with a fine moderate flame. The test

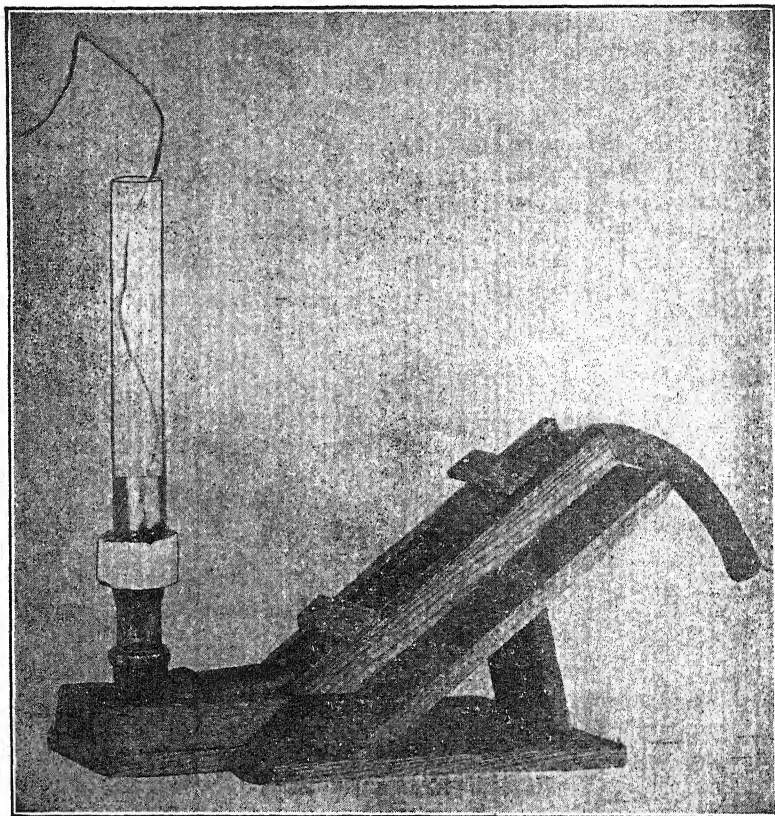


FIG. 5.—Suggested Means for Holding Porcelain Dielectric Strength Test Specimen.

(19.05 mm.) in diameter, placed exactly opposite each other.

NOTE.—A suggested means for holding the specimen, using mercury electrodes, is shown in Fig. 5. This consists of a metal tube supported in a frame. The specimen is affixed to the metal tube using ordinary sealing wax, and a short glass tube is similarly affixed to the upper surface of the specimen.

specimen should never be heated when affixing the tubes, as the heat may set up strains in the porcelain specimen that will materially affect its properties.

Mercury is introduced into both tubes, avoiding entrapped air, and lead wires are placed in the mercury. The entire set-up is immersed in high-grade mineral insulating oil. It is best to have the ends of the tubes projecting above the oil level.

(b) The testing voltage shall be increased at a constant rate of approximately 1000 v. per sec. until puncture occurs.

(c) Not less than five specimens shall be punctured at a normal room temperature of about 20 C. (68 F.).

### Report

22. The report shall include the following:

(1) The breakdown voltage for each specimen in kilovolts,

(2) The thickness of each specimen in mils at point of break,

(3) The dielectric strength in volts per mil calculated from the values reported in Items (1) and (2), and

(4) The average breakdown voltage, the average thickness of the specimen at break, and the average dielectric strength in volts per mil for the specimens tested.

### EFFECT OF HEAT

#### *Method A. Resistance to Thermal Change*

### Apparatus

23. The apparatus shall consist of a hot-water bath maintained at 100 C. (212 F.) and a cold-water bath maintained at 0 C. (32 F.).

### Test Specimens

24. The test specimens shall be as described in Section 13.

### Procedure

25. (a) The test specimen shall be immersed in the cold-water bath for 10 min. and then transferred as quickly as possible to the hot-water bath, and allowed to remain there for 10 min. The specimen shall be transferred back to the cold-water bath and the cycles continued until the specimen breaks.

(b) Not less than five specimens shall be tested.

### Report

26. The number of cycles necessary to cause fracture of each test specimen shall be reported.

#### *Method B. Effect on Flexural Strength<sup>4</sup>*

### Apparatus

27. A furnace in which a temperature of 900 C. (1652 F.) can be obtained and any suitable testing machine for determining the flexural strength of small beams may be used.

### Test Specimens

28. The test specimens shall be as described in Section 13.

### Procedure

29. (a) Not less than 12 specimens shall be tested.

(b) One half of the specimens shall be placed in the furnace and the rate of heating so adjusted that the temperature reaches 900 C. (1652 F.) in 2 hr. The furnace shall then be allowed to cool at such a rate that room temperature is reached in 4 hr. Each of the specimens shall then be subjected to a flexure test by placing them on supports 5 in. apart and loading them at the center.

(c) The other half of the specimens shall be subjected to the flexure test by placing them on supports 5 in. apart and loading them at the center.

### Report

30. The report shall include the following:

(1) The load in pounds required to break each specimen that was subjected to heat treatment, together with the average,

(2) The load in pounds required to break each specimen not subjected to

<sup>4</sup> This method is particularly suited to testing porcelain for spark plugs and heating devices, but may be used for testing other porcelains.



heat treatment, together with the average, and

(3) The percentage loss of flexural strength due to heat treatment, calculated from the two averages.

### POROSITY

#### Scope

31. (a) In the case of relatively coarse and porous bodies, the porosity of porcelain may be indicated by the amount of water which they absorb, expressed as percentage (by weight) water absorption. Method A, described in Sections 32 to 35, is intended to be used for such cases and shall be used whenever the value of water absorption is 0.1 per cent or over.

(b) In the case of the more dense porcelain bodies, the porosity of porcelain may be indicated by the amount of gas contained in a sample of known volume, expressed as percentage (by volume) pore volume. Method B, described in Sections 36 to 41, is intended to be used for such cases and shall be used whenever the value of water absorption (method A) is less than 0.1 per cent.

(c) Method C, described in Sections 42 to 46, covers the procedure for determining porosity as indicated by the penetration of an alcohol-dye solution into the porcelain body.

#### *Method A. Water Absorption*

##### Apparatus

32. The apparatus shall consist of the following:

(a) *Balance*.—A chemical balance.

(b) *Oven*.—An oven capable of maintaining a temperature of  $120 \pm 5$  C. ( $248 \pm 9$  F.).

##### Test Specimens

33. Each test specimen shall weigh from 2 to 50 g. and shall have at least 25 per cent of the surface newly fractured.

#### Procedure

34. (a) The specimens shall be dried for 2 hr. in the oven at 120 C. (248 F.) and then cooled in a desiccator to the approximate room temperature. Each specimen shall be weighed immediately upon removal from the desiccator.

(b) The specimen shall then be transferred to a beaker of briskly boiling distilled water and allowed to remain totally submerged therein for 30 min. It shall then be removed and quenched in distilled water at room temperature (about 20 C. (68 F.)) The surface water shall be wiped off with a soft cloth and the specimen weighed immediately.

(c) Not less than five specimens shall be tested.

#### Report

35. The report shall include the following:

(1) The dry weight of each specimen in grams,

(2) The weight of each specimen after immersion in grams,

(3) The percentage water absorption of each specimen, and

(4) The average percentage water absorption of all specimens tested.

#### *Method B. Pore Volume*

##### Pore Volume

36. The pore volume of porcelain shall be determined by measuring (at atmospheric pressure) the volume of air contained in a sample of porcelain of known volume.

##### Apparatus

37. (a) Any suitable apparatus based on the laws of gas expansion which will accomplish the measurement specified in Section 36 may be used. The McLeod gage type of porosimeter is especially adaptable to this determination. This apparatus consists of a receptacle and cap fitted together by a ground airtight joint. The cap is surmounted by a calibrated capillary tube and stopcock.

The specimen is placed in the receptacle and prevented from rising into the cap by means of a constriction or pin. The receptacle is connected by a glass or metal pipe at the bottom to a heavy rubber hose which in turn is connected to a glass leveling bulb. The total

(b) *Volume and Length of Capillary.*—For porcelain of low-pore volume it is necessary to use a capillary of small diameter to read the volume of accumulated air accurately. Thus, for porcelain up to 1 per cent pore volume the effective volume of the capillary shall be at

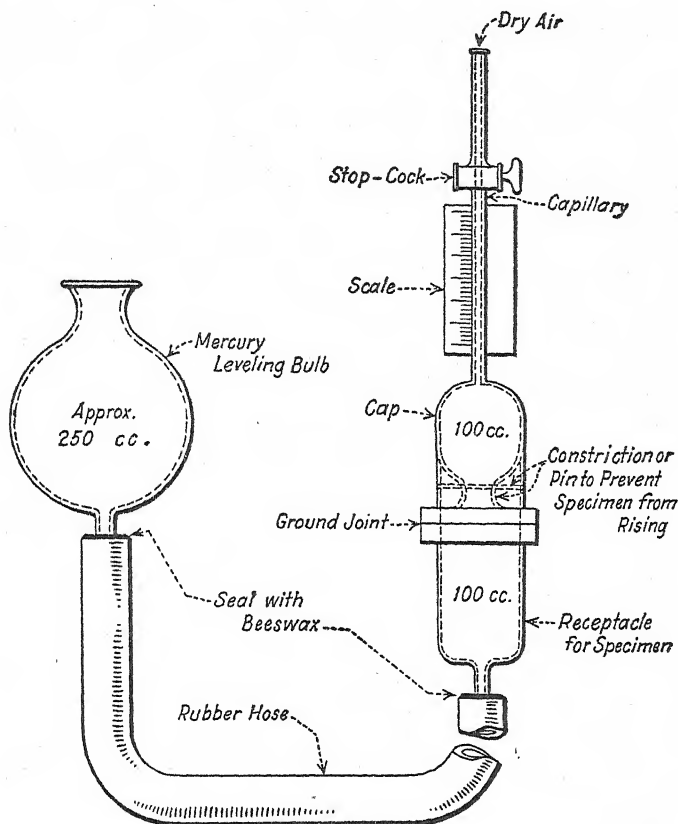


FIG. 6.—Diagrammatic Sketch of McLeod Porosimeter.

volume of the specimen receptacle and cap should be approximately 200 ml. Figure 6 shows a diagrammatic sketch of a McLeod gage-type porosimeter.

NOTE.—Suitable porosimeters of the McLeod gage type are: (1) glass apparatus of Washburn and Bunting,<sup>5</sup> and (2) metal apparatus of Navias.<sup>6</sup>

least 0.5 ml. (for a specimen of 50 ml. volume). For porcelain having a higher pore volume than 1 per cent, either a longer capillary or a capillary of larger diameter is required.

(c) *Atmospheric Conditions.*—It is essential that the determination be made using dry air. This may be provided by any suitable means.

<sup>5</sup> Edward W. Washburn and Elmer N. Bunting, "The Determination of the Porosity of Highly Vitriified Bodies," *Journal, Am. Ceramic Soc.*, Vol. 5, pp. 527-535 (1922).

<sup>6</sup> Louis Navias, "Metal Porosimeter for Determining the Pore Volume of Highly Vitriified Ware," *Journal, Am Ceramic Soc.*, Vol. 8, pp. 816-821 (1925).



### Test Specimen

38. The test specimen shall consist of two or more pieces of porcelain having a total volume of not less than 50 ml. At least 50 per cent of the surface of the parts used shall be newly fractured.

### Procedure

39. The following procedure is based on the use of a McLeod gage type of porosimeter:

(a) The fractured pieces of porcelain shall be placed in the receptacle, the ground joint greased, and the cap secured in position so that the apparatus is airtight.

(b) Before determinations are made, any moisture entrapped on the inner surfaces of the porosimeter or which may be on the test specimen shall be removed. To do this, the leveling bulb shall be raised until the mercury in the capillary is above the stopcock. The stopcock shall then be closed and the leveling bulb lowered so that the test specimen is subjected to a vacuum for at least 1 min. Any moisture inside the porosimeter then vaporizes and by again raising the leveling bulb the gas is collected in the capillary and shall be finally expelled by opening the stopcock.

(c) Immediately after removing the moisture the leveling bulb shall be lowered, exposing the test specimen to the air for at least 1 min. The leveling bulb shall again be raised until the mercury in the capillary is above the stopcock. The stopcock shall then be closed and the leveling bulb lowered so as to expose the entire specimen to the vacuum for at least 1 min. The air contained in the porcelain then expands into the evacuated space around and above the porcelain. The mercury bulb shall then be raised again and the air collected in the capillary tube. When the mercury surface in the leveling bulb is on a level with the mercury meniscus

in the capillary, the accumulated air in the capillary is at atmospheric pressure and its volume,  $v$ , shall be read. This shall be considered to be the pore volume.

(d) Several determinations of the pore volume should immediately be made as described in Paragraph (c). The values of pore volume thus obtained should be practically equal, and their average may be taken as the pore volume of the specimen,  $v$ . Lack of agreement among these values may be traced to leaks or moisture in the apparatus.

NOTE.—To test for leaks in the apparatus: After taking a reading, expel all gas from the apparatus. Close the stopcock. Raise and lower the leveling bulb several times. The level of the mercury in the capillary should rise to the stopcock when the leveling bulb is raised.

(e) The total volume,  $V$ , of the pieces of each specimen shall be measured to the nearest 0.2 ml. using any suitable method.

(f) Not less than five specimens shall be tested.

### Calculations

40. The percentage of pore volume shall be calculated as the volume of gas contained in the total volume of porcelain:

$$\text{Percentage of pore volume} = \frac{v \times 100}{V}$$

where:

$v$  = pore volume as determined in Sections 39 (c) and (d), and  
 $V$  = total volume of test specimen as determined in Section 39 (e).

### Report

41. The report shall include the following:

(1) The percentage of pore volume of

each specimen, the average of these values, and the percentage average deviation from the mean,

(2) The total volume of each test specimen,

(3) The name and type of the apparatus used, and

(4) The total volume of the specimen receptacle.

#### *Method C. Dye Penetration*

##### **Apparatus**

42. The apparatus shall consist of a suitable pressure chamber of such dimensions as to accommodate the test specimen when immersed in the dye solution with arrangements for obtaining and maintaining the required pressure for the required time.

##### **Reagent**

43. *Fuch sine Solution.*—A solution consisting of 1 g. of basic fuch sine in 1 liter of 50 per cent alcohol is suitable. If a denatured alcohol is used, one should be selected which does not react with the dye to cause fading of the color.

##### **Test Specimens**

44. The test specimens shall be freshly broken fragments of the porcelain body, having clean and apparently unshattered surfaces exposed. At least 75 per cent of the area of such specimens should be free from glaze or other surface treatment. Fragments approximately  $\frac{1}{4}$  in. in the smallest dimension up to  $\frac{3}{4}$  in. in the largest dimensions are recommended.

##### **Procedure**

45. (a) The test specimens shall be placed in the pressure chamber and

completely immersed in the fuch sine solution.

(b) A pressure of approximately 4000 psi  $\pm$  10 per cent shall be applied for approximately 15 hr. or an optional pressure of 10,000 psi.  $\pm$  10 per cent for 6 hr. may be used.

(c) At the conclusion of the application of test pressure, the specimen shall be removed from the pressure chamber, thoroughly dried, and broken as soon as possible for visual examination.

(d) Porosity is indicated by penetration of the dye into the porcelain body to an extent visible to the unaided eye. Penetration into small fissures formed in preparing the test specimen shall be disregarded.

##### **Report**

46. The report shall include a statement of the observations recorded in accordance with the examination prescribed in Section 45 (d).

#### **POWER FACTOR AND DIELECTRIC CONSTANT**

##### **Procedure**

47. The power factor and dielectric constant shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

#### **RESISTIVITY**

##### **Procedure**

48. The resistivity shall be determined in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257).<sup>3</sup>

## *Standard Methods of*

### TESTING STEATITE USED AS ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 667 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 667; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These methods outline procedures for making the various tests described when they are required for an investigation or examination of steatite used as electrical insulation. They are not to be interpreted as forming test specifications for complete insulators.

NOTE.—The glaze on a steatite part may affect certain properties of the material, depending not only upon its presence but also upon its thickness and composition. While these methods were developed for testing unglazed specimens, they are equally suitable for glazed specimens. The report should in all cases indicate whether the specimens tested were glazed or unglazed.

#### POWER FACTOR AND DIELECTRIC CONSTANT

##### Procedure

2. Power factor and dielectric constant shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1942 to 1944.

#### RESISTIVITY

##### Procedure

3. Resistivity shall be determined in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257) of the American Society for Testing Materials.<sup>3</sup>

#### DIELECTRIC STRENGTH

##### Procedure

4. Dielectric strength shall be determined in accordance with the procedure described in Sections 19 to 22 of the Standard Methods of Testing Electrical Porcelain (A.S.T.M. Designation: D 116) of the American Society for Testing Materials.<sup>3</sup>

#### POROSITY

##### Procedure

5. Porosity shall be determined in accordance with either of the procedures described in Sections 31 to 41 of Standard Methods D 116.<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## IMPACT RESISTANCE

## Procedure

6. Resistance to impact shall be determined in accordance with the simple beam (Charpy type) test as described in Sections 7 to 11 of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S. T.M. Designation: D 256) of the American Society for Testing Materials,<sup>3</sup> and the test specimen shall be the same as specified for ceramic materials.

## FLEXURAL STRENGTH

## Procedure

7. Flexural strength shall be determined in accordance with the procedure described in Sections 12 to 17 of Standard Methods D 116, except that the test specimens shall be 6 in. (15.2 cm.) in length and 0.5 in. (1.27 cm.), 0.75 in. (1.91 cm.), or 1.125 in. (2.86 cm.) in diameter, whichever value is comparable to that of the finished product.

## COMPRESSIVE (CRUSHING) STRENGTH

## Apparatus

8. Any universal testing machine of suitable capacity may be used provided the error in the loading range does not exceed 1 per cent.

## Test Specimens

9. The compression test specimens shall be in the form of cylinders and shall be 0.75 in. (1.91 cm.) in diameter and 1.125 in. (2.86 cm.) in length. The flat surfaces shall be ground smooth and parallel.

## Procedure

10. (a) The diameter and length of each specimen shall be measured with a micrometer graduated to 0.001 in. (0.025

mm.). The value of the diameter to be used in the calculations shall be the average of two measurements taken at right angles.

(b) A contact pad or cushion of  $\frac{1}{8}$ -in. soft steel (Rockwell hardness B65, max.) shall be placed between the upper and lower faces of the specimen and the heads of the testing machine to equalize irregularities in the surfaces. Fresh cushions shall be used for each specimen.

(c) The test shall be made at a temperature of approximately 20 C. (68 F.).

(d) The crosshead speed of the testing machine shall be such that the load can be accurately weighed.

(e) Not less than five specimens shall be tested.

(f) *Initial and Ultimate Failure.*—Note shall be made of the load in pounds at initial failure, but the value of the load in pounds at ultimate failure shall be determined and used in calculating the compressive (crushing) strength.

## Report

11. The report of the compression test shall include the following:

(1) The diameter and length of each specimen,

(2) The load in pounds on each specimen at initial failure and the load in pounds at ultimate failure,

(3) The compressive (crushing) strength of each specimen in pounds per square inch,

(4) The average of the results reported in Item 3,

(5) The rate of application of the load,

(6) The name and rating of the testing machine used in making the tests, and

(7) A statement as to the behavior of the material while under load.

# Standard Methods of

## TESTING SOLID FILLING AND TREATING COMPOUNDS USED FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 176 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 176; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover physical and electrical tests for solid filling and treating compounds for electrical insulation.

NOTE.—Before testing under these methods it may be necessary to degas materials that tend to entrap gases (usually due to high viscosity) or to froth on heating or both. (See Section 4 (b).)

### PHYSICAL TESTS

#### General Physical Tests

2. Each of the following tests shall be made in accordance with the method of test applicable, of the American Society for Testing Materials, as indicated:

(a) *Melting Point*.—Standard Method of Test for Melting Point of Petrolatum (A.S.T.M. Designation: D 127).<sup>3</sup>

(b) *Softening Point* (for compounds having no definite melting point).—Tentative Method of Test for Softening Point (Ball and Shouldered Ring Apparatus) (A.S.T.M. Designation: E 28).<sup>4</sup>

(c) *Flash and Fire Points*.—Standard

Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>3</sup>

(d) *Evaporation*.—Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).<sup>3</sup>

(e) *Viscosity* (Temperature range from melting point to 150 C.).—Saybolt Furol, Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>3</sup>

(f) *Penetration* (Temperature range from melting or softening point to -25 C.).—Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).<sup>3</sup>

### BRITTLINESS

#### Procedure

3. To determine the brittleness of the material, the compound shall be melted and poured on a piece of sheet metal in a layer approximately  $\frac{1}{16}$  in. in thickness and then submerged in water having a temperature of 70 F. (The melting and pouring temperature shall not exceed by more than 20 F. the softening point (ball and shouldered ring method as determined under Section 2 (b)). It shall then be cooled 5 F. and held at this temperature for 5 min. The cooling

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1923 to 1944, being revised in 1924, 1925, 1929, 1933, 1934, 1935, 1936, 1937, 1940, and 1942.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall then be continued in increments of 5 F. At each temperature interval the compounds shall be tested with the blade of a knife, inserting the point with the blade as nearly parallel with the sheet metal as is practicable and pulling the compound from the sheet metal as rapidly as possible by rotating the blade about its back as an axis. The temperature at which the compound no longer stretches but snaps shall be considered the breaking point.

### SPECIFIC GRAVITY

#### Purpose

4. (a) The purpose of this test is to determine the specific gravity of both untreated and degassed compounds. The approximate volume occupied by entrapped gas can be calculated from the difference in specific gravity of an untreated sample and a degassed sample.

(b) If degassing is required, it shall be performed by heating the material in a vacuum oven. The temperature and vacuum shall be high enough and the time long enough to insure driving off of mechanically entrapped gases but should tend to decompose the compounds as little as possible.

#### Water Displacement Methods

#### Procedure

5. The specific gravity shall be determined by the Standard Method of Test for Specific Gravity of Asphalts and Tar Pitches Sufficiently Solid to be Handled in Fragments (A.S.T.M. Designation: D 71)<sup>3</sup> or by the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70)<sup>3</sup> of the American Society for Testing Materials.

#### Plummet Displacement Method

#### Scope

6. The specific gravity of the ma-

terial at the pouring temperature is calculated from the weight of compound displaced by a calibrated aluminum plummet.

#### Apparatus

7. The apparatus shall consist of the following:

(a) *Balance*.—An analytical balance equipped with pan straddle.

(b) *Plummet*.—An aluminum plummet of suitable shape weighing 5 to 10 g.

(c) *Beaker*.—A 400-ml. glass<sup>5</sup> beaker wrapped with asbestos.

(d) *Thermometer*.—A thermometer of suitable range.

(e) *Wire*.—Two pieces of fine copper wire.

#### Procedure

8. (a) *Calibration of Plummet*.—The following weight determinations of the plummet shall be made to the nearest 1 mg.:

$a$  = weight in air, and

$b$  = weight suspended in water at 25 C. (77 F.).

Then:

$$a - b = \text{weight of water displacement at 25 C. (77 F.)}$$

The value of the plummet displacement ( $D_{t_p}$ ) in terms of grams of water at 25 C. (77 F.) shall then be corrected to the pouring temperature,  $t_p$ , in Centigrade degrees by means of the following formula:

$$D_{t_p} = 0.000076^a (t_p - 25)(a - b) + (a - b)$$

(b) *Testing of the Sample*.—The sample shall be carefully melted in the beaker and the temperature raised to approximately 15 C. (27 F.) above the desired test temperature. The beaker shall be placed on the straddle and the plummet suspended in the compound by the fine copper wire. (The weight of the wire should be balanced by placing

<sup>a</sup> Coefficient of cubical expansion of aluminum per degree Centigrade.

<sup>5</sup> Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

an equal weight of the wire on the opposite balance pan.)

The scales shall be balanced approximately and at the same time the sample shall be stirred slowly, using the thermometer as a stirring rod. When the sample has cooled to the desired temperature, the weighing shall be completed rapidly.

(c) *Calculation of Specific Gravity,  $t_p/25$  C.*—The specific gravity shall be calculated as follows:

$$\text{Sp. gr., } t_p/25 \text{ C.} = \frac{W_a - W_c}{D_{t_p}}$$

where:

$W_a$  = weight of plummet in air, and

$W_c$  = weight of plummet in compound.

#### *Volume of Entrapped Gases*

##### Calculation

9. The percentage of entrapped gases may be calculated as follows:

$$V = \frac{b - a}{b} \times 100$$

where:

$V$  = volume of entrapped gases in per cent,

$b$  = specific gravity of degassed compound, and

$a$  = specific gravity of untreated compound.

##### COEFFICIENT OF EXPANSION

NOTE.—Certain materials when cooled form voids within the mass, and no satisfactory method has been found to measure their coefficient of expansion.

##### Purpose

10. The purpose of this test is to determine the true and effective coefficients of expansion of compounds. The true coefficient of expansion is determined on compounds which are free of entrapped gases. The effective coefficient of expansion is determined on materials which

have not been degassed just prior to test. It is important for many purposes to know the effective coefficient of the material as received or after heating to the maximum temperature of application. Consistent results, however, may only be obtained with gas-free compounds.

##### Scope

11. The following five methods are covered:

*Methods A and B.*—Methods *A* and *B* for true coefficient of expansion are intended for use only where the uniformity of the material under test justifies a high degree of precision. Method *A* is suitable for testing low-viscosity types such as waxes and petrolatums. Method *B* is suitable for testing asphalts and high-viscosity materials, also for opaque materials which may give difficulty in reading the glass scale of Method *A*.

*Methods C, D, and E.*—Methods *C*, *D*, and *E* are intended for faster testing where high precision may not be justified. These methods may be used for determining either true or effective coefficient of expansion but not as referee methods.

#### *Method A. Using Glass Flask*

##### Apparatus

12. The apparatus shall consist of the following:

(a) *Flask.*—A glass flask<sup>6</sup> holding approximately 250 ml. to the zero mark, and graduated for 25 ml. in 0.1-ml. divisions, the neck of the flask being 1 cm. in internal diameter.

(b) *Oil Bath.*—For heating the sample, a cylindrical oil bath approximately 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth, with a false bottom 1 in. from the bottom and provision for circulating and heating the oil.

<sup>6</sup> A Pyrex or quartz glass flask is very satisfactory for this purpose.

(c) *Metal Collar*.—Lead or iron collars for use on the neck of the flask during test to prevent oil currents of the bath from moving the flask.

### Calibration

13. The capacity of the flask at the zero point and several points on the scale shall be determined by filling the flask with distilled water at a known temperature and weighing.

### Procedure

14. (a) The flask shall be maintained under a vacuum of 25 in. of mercury and a temperature 50 C. higher than the softening point (ball and shouldered ring method, as determined under Section 2 (b)) while being filled and for approximately 30 min. after filling is complete. The flask shall be filled to within the last milliliter marked on the neck when held at the maximum test temperature and slowly cooled to room temperature (10 to 12 hr.). Before starting the test, the flask shall be examined for the presence of cavities or irregular contraction of the compound. Some compounds, after cooling below the liquid state, tend to stick to the sides of the neck of the flask. In such cases it is necessary to gradually warm the neck and flow the compound to meet the rest, after which the flask shall be placed in the bath for several hours to insure temperature equilibrium.

(b) With the compound satisfactorily placed in the flask at the lowest temperature, the height of the column in the neck shall be read and the bath then slowly heated. Readings shall be taken at 50 C. intervals, holding the bath as constant as possible at each point until no more expansion occurs at that point, the procedure being repeated for each point until maximum temperature is reached.

(c) *Precautions*.—During the test,

temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of the expansion of the compound shall be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction it may be reduced to 30 min.

### Calculation

15. (a) After securing the readings over the temperature range desired, a curve shall be plotted from the temperature and expansion readings from which the coefficient of expansion shall be calculated as follows:

$$E = \frac{V_1 - V}{(T_1 - T)V} + C$$

where:

$E$  = coefficient of expansion of the compound,

$V$  = the original volume occupied by the compound,

$V_1$  = volume at higher temperature occupied by the compound,

$T$  = original temperature,

$T_1$  = higher temperature, and

$C$  = a constant = coefficient of expansion of glass container.

(b) The coefficient of expansion shall be calculated for three temperature ranges, as follows:

(1) From the minimum temperature at which the measurement was made to 10 C. below the softening point. This is intended to give the average coefficient for the solid condition.

(2) From 5 C. above the softening point to 100 C. This is intended to give the average coefficient for the liquid condition.

(3) From the minimum temperature at which a measurement was made to 100 C.



**Report**

16. The report shall include the following:

- (a) Type of cell used,
- (b) Copy of the volume-temperature curve,
- (c) Temperature ranges as defined under Section 15 (b), and
- (d) Coefficient of expansion corresponding to each of the three temperature ranges.

*Method B. Using Metallic Cell***Apparatus**

17. The apparatus shall consist of the following:

(a) *Metal Cell*.—A cell made of steel, consisting of four parts, a cylinder about 2.5 in. in internal diameter having a rigid bottom, a metallic gasket, a cover to which a steel capillary tube is attached. The cell shall have an internal volume of approximately 250 ml. A metallic cell that has been found suitable is described in Appendix I.

(b) *Oil Bath*.—An oil bath as described in Section 12 (b), method A, with the exception that provision shall be made for supporting the metal cell.

**Calibration**

18. The cell shall be calibrated to determine its volume at various temperatures as follows:

(a) Weigh the assembled cell to determine its tare weight.

(b) Fill the cell with mercury until replacing the cover causes some to extrude through the capillary tubing. Record the weight of the cell and mercury and note the temperature.

(c) Place cell in the oil bath in an inverted position. The capillary tubing should extend over the side of the oil bath in such a way that the extruded mercury may be caught in a beaker.

The oil bath which is several degrees above room temperature causes some mercury to be extruded from the capillary tube. When all expansion has taken place, weigh the mercury collected.

(d) Adjust the oil bath for other test temperatures and note the amounts of mercury extruded. The weight of mercury in the cell at any temperature is thus determined and the volume may be calculated.

**Procedure**

19. (a) While filling the cell it shall be placed in an oil bath and maintained at a temperature 50 C. higher than the softening point of the compound (ball and shouldered ring method, as determined under Section 2 (b)). When the cell has been filled to within  $\frac{1}{4}$  in. of the cover it shall be placed in a vacuum oven and maintained at a vacuum of 25 in. of mercury and a temperature 50 C. higher than the softening point of the compound for a period of not less than 30 min. nor more than 45 min. At the end of this period the cell shall be slowly cooled to room temperature, and any irregularities in the surface of the compound removed.

(b) The cover shall then be screwed on and the cell and compound weighed again.

(c) Sufficient mercury shall be poured into the cell so that some is extruded when the cover is screwed down. The cell shall then be weighed again.

(d) The cell shall then be inverted and placed in the oil bath, and the procedure outlined under calibration, Section 18 (c) and (d), again followed for 5 C. intervals.

(e) *Precautions*.—Only clean, distilled mercury shall be used. During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of the expansion of the compound should be made at

intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction, it may be reduced to 30 min.

### Calculation

20. (a) After volumetric determinations have been made over the desired temperature range, a curve shall be plotted between volume and temperature readings from which the coefficient of expansion shall be calculated as follows:

$$E = \frac{V_1 - V}{(T_1 - T) V}$$

where:

$E$  = coefficient of expansion of the compound,

$V$  = original volume occupied by the compound,

$V_1$  = volume at higher temperature occupied by the compound,

$T$  = original temperature, and

$T_1$  = higher temperature.

(b) The coefficient of expansion shall be calculated for the same three ranges as prescribed in method A, Section 15 (b).

### Report

21. The report shall include the following:

(a) Type of cell used,

(b) Copy of the volume-temperature curve,

(c) Temperature ranges as defined under Section 15 (b), and

(d) Coefficient of expansion corresponding to each of the three temperature ranges.

### *Method C. Specific Gravity Method*

#### Procedure

22. The specific gravity of untreated

or degassed compounds at two test temperatures may be determined by one or more of the procedures specified in Sections 5 to 8 applying to the state of the materials at the temperatures between which measurements are desired.

NOTE.—When the temperature range includes the range over which the material changes from solid to liquid, a true coefficient of expansion cannot be calculated, although for practical purposes this may be done.

### Calculation

23. From the temperature and specific gravity readings the coefficient of expansion shall be calculated as follows:

$$E = \frac{\text{sp. gr. at } T - \text{sp. gr. at } T_1}{(T_1 - T) \text{ sp. gr. at } T_1}$$

where:

$E$  = coefficient of expansion of the compound,

$T$  = initial temperature, and

$T_1$  = higher temperature.

### Report

24. The report shall include the following:

(a) Method used,

(b) Test temperatures, and

(c) Coefficient of expansion over temperature range used.

### *Method D. Pycnometer Contraction*

#### Scope

25. This method is a modification of the specific gravity method (method C). It may be applied to either untreated or degassed compounds.

#### Apparatus

26. The apparatus shall consist of the following:

(a) *Flask*.—A 100-ml. volumetric glass<sup>5</sup> flask having the zero mark as near as possible to the bulb of the flask.

(b) *Oil Bath*.—An oil bath which may

consist of a tall-form glass<sup>5</sup> beaker of sufficient size so that when the flask is supported about 1 in. from the bottom the oil level will reach at least to the zero mark of the flask.

(c) *Metal Collar*.—Lead or iron collars for use on the neck of the flask during heating to prevent oil currents of the bath from moving the flask.

### Procedure

27. (a) The flask shall be weighed, and then filled to within approximately 10 ml. of the zero mark with the compound to be tested, care being taken that at no time shall the temperature of the compound exceed the softening point (ball and shouldered ring method, Section 2 (b)) for use by more than 10 C. Care shall also be taken so that none of the compound remains in the stem of the flask. The flask shall be cooled to room temperature, weighed, immersed in the oil bath, and placed in an oven previously heated to the maximum temperature at which the test is to be conducted. After the oil bath reaches the test temperature, it shall be allowed to remain at this temperature for 1 hr. The flask shall be removed from the oven, and mercury from a burette shall be quickly poured into it until the level of the compound is within 0.5 ml. of the zero mark. The quantity of mercury used shall be noted in milliliters. The flask shall be replaced in the oven for 20 min., removed again, and the mercury titration shall be continued until the level of the compound is exactly at the zero mark. The number of milliliters of mercury used in both titrations shall be recorded.

(b) The oven temperature shall be reset at the next lower test point, temperature equilibrium obtained as above, and the titration shall be repeated. The same procedure shall be continued at all subsequent test points, the milliliters of

mercury added at each point being recorded.

### Calculations

28. To find the coefficient of expansion of the material between any two temperatures, the calculations shall be made as follows:

$$V = A + 0.000182 A (t - 20)$$

where:

$V$  = volume of mercury  $A$  at room temperature corrected to temperature  $t$ , and

$A$  = volume of mercury at room temperature.

The coefficient of cubic expansion of mercury is taken as 0.000182, that of glass as 0.000025.

*Example*.—If volume of flask is 100 ml. at 20 C.; volume at  $t = 100 + 0.0025 (t - 20)$  ml.

Let  $X = V_1 - M_1$  = vol. of compound at  $T_1$ ,  
Let  $Y = V_2 - M_2$  = vol. of compound at  $T_2$ ,

Then:

$$\begin{aligned} \text{The expansion in cu. cm.} \\ \text{per gram, from } T_1 \text{ to } T_2 = \frac{X}{W} - \frac{Y}{W} \end{aligned}$$

$$\begin{aligned} \text{The expansion per} \\ \text{cu. cm., from } T_1 \text{ to } T_2 = \frac{\frac{X - Y}{W}}{\frac{Y}{W}} = \frac{X - Y}{Y} \end{aligned}$$

$$E = \frac{X - Y}{Y (T_1 - T_2)}$$

where:

$E$  = coefficient of expansion in cubic centimeters per cubic centimeter, per degree Centigrade,

$W$  = weight of sample in grams,

$T_1$  = higher temperature in degrees Centigrade,

$T_2$  = lower temperature in degrees Centigrade,

$M_1$  = milliliters of mercury (corrected) added at  $T_1$ .

$M_2$  = sum of milliliters of mercury  
(corrected) added at  $T_1$  and  
at  $T_2$ ,

$V_1$  = corrected volume of flask at  $T_1$ ,  
and

$V_2$  = corrected volume of flask at  $T_2$ .

To obtain the curve of expansion, plot the points  $\frac{W}{X}$ ,  $\frac{W}{Y}$ , etc., which are the densities at various temperatures.

### Report

29. The report shall include the following:

- (a) Method used,
- (b) Copy of curve of expansion,
- (c) Temperature ranges used, and
- (d) Coefficient of expansion over each of the temperature ranges.

#### *Method E. Pycnometer Expansion*

### Scope

30. This method is another modification of the specific gravity method (Method C) and may also be applied to either untreated or degassed materials.

### Apparatus and Calibration

31. The pycnometer and bath required are the same as described in Section 26, except that the neck of the flask shall be cut off at the 100-ml. point and ground square. A metal pycnometer may be used provided its coefficient of expansion is known and is applied in the calculations (Section 33).

### Procedure

32. In this procedure the pycnometer shall be allowed to cool slowly to the lowest test temperature. During the cooling period the flask shall be kept filled by adding more compound, and after equilibrium is reached the excess material shall be removed by passing a sharp, flat blade over the rim. The

flask shall then be removed from the bath and quickly weighed. Knowing the tare weight and volume of the flask, the specific gravity may be determined. For successively higher temperatures, it is only necessary to weigh the extruded portion.

### Calculations

33. From the temperature and weight readings the coefficient of expansion may be calculated as follows:

$$E = \frac{W - W_1}{W_1 (T_1 - T)} - \frac{WC}{W_1}$$

where:

- $E$  = coefficient of expansion,  
 $W$  = initial weight of the compound in the flask,  
 $W_1$  = weight of the compound in the flask at higher temperature,  
 $T$  = initial temperature,  
 $T_1$  = higher temperature, and  
 $C$  = coefficient of expansion of the flask.

### Report

34. The report shall include the following:

- (a) Method used,
- (b) Test temperature, and
- (c) Coefficient of expansion over temperature ranges used.

## ELECTRICAL TESTS DIELECTRIC STRENGTH

### Purpose

35. The purpose of this test is to determine the short-time dielectric strength of solid filling and treating compounds. It is of value for purposes of comparison or as an indicator of the condition of a compound, but it is not a measure of the dielectric strength of compounds when subjected to prolonged stresses.

## Apparatus

36. (a) The equipment used for producing, controlling, and measuring the test voltage shall conform to that prescribed in Section 3 of the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>4</sup>

(b) *Electrodes*.—The compound shall be tested between polished hemispherical electrodes  $\frac{1}{2}$  in. in diameter separated by a gap of 0.1 in.

NOTE.—A convenient form of apparatus for holding the electrodes and compound is described in Appendix II. It is recognized that when liquid materials are being tested, this method is inconsistent with the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117) of the American Society for Testing Materials.<sup>4</sup> However, since spherical electrodes are considered preferable for solid and semisolid materials, the advantage of using spherical electrodes, in order that direct comparison can be made in both the liquid and solid states, is sufficiently great to justify the inconsistency with Methods D 117.

## Procedure

37. (a) A representative sample of the material shall be taken from the original package. This sample shall be melted and poured directly into the testing container, care being taken not to overheat the compound nor to entrap air. A melting and pouring temperature of approximately 50 C. above the softening point (ball and shouldered ring method) as determined under Section 2 (b) is recommended. Pasteboard test receptacles shall be thoroughly dried by heating before using, and the entire test container assembly shall be heated to its safe maximum temperature (85 to 90 C. if phenolic insulation is used) before the compound is poured into it.

(b) The testing container, after filling,

shall be placed in a suitable temperature control chamber and left for 4 hr. or until the compound has been brought uniformly to the desired test temperature. The temperature may be determined by a thermocouple inserted along the axis of one of the electrodes to a point near the tip.

(c) Voltage shall then be applied to the test specimen at a uniform rate of increase of 1000 v. per sec. from zero to breakdown.

(d) Specimens shall be tested in air unless the breakdown voltage exceeds 100 kv. If the breakdown voltage is greater than 100 kv., the test container shall be immersed in clean, dry oil. Before the specimen is immersed, the oil shall be brought to the test temperature. Care shall be taken not to contaminate the compound with the oil.

(e) Variations of gap setting with temperature may be determined by varying the temperature of the test assembly when empty. For well-designed test apparatus, these variations are so small as to be negligible. For maximum accuracy, or for cases where the variation might be appreciable, a temperature correction curve for the gap may be determined and allowance made for gap variation when a test setting is made.

## Number of Tests

38. (a) As the dielectric strength of filling and treating compounds varies widely with temperature, it is recommended that tests be made at a sufficient number of temperatures to establish the dielectric strength-temperature curve from 0 to 100 C. For this purpose, tests should be made at 0, 30, 70, and 100 C.

(b) Five specimens shall be tested at each temperature and the average value taken as the short-time dielectric

strength of the compound at that temperature.

### Report

39. The report shall include the following:

(a) The short-time dielectric strength-temperature curve, and

(b) The individual values of the breakdown voltage.

### VOLUME RESISTANCE-TEMPERATURE CHARACTERISTICS

#### Purpose

40. The purpose of this test is to determine the volume resistance - temperature characteristics of solid filling and treating compounds. It is of value as a comparative test and also may be used in determining the volume resistance on an ohm-centimeter basis.

#### Temperature Range

41. The volume resistance of this class of material varies with the temperature, generally decreasing rapidly with increase of temperature. It is, therefore, recommended that a sufficient number of tests be made at different temperatures to establish the volume resistance - temperature curve. To be of most significance, the test shall include determinations made up to the highest service temperatures. At room temperatures and below, the volume resistance of practically all of these compounds is so high that it cannot be measured with the ordinary galvanometer equipment.

#### Apparatus

42. A suitable conductivity cell consisting of parallel planes, concentric cylinders, or coaxial cones shall be used in determining the volume resistance of the compound. The distance between electrodes shall be not less than 0.75 mm.

(0.03 in.) nor more than 5 mm. (0.2 in.). The voltage gradient shall not exceed 200 v. per millimeter (5 v. per mil). The area of the electrode shall be sufficiently large so that the current flow can be measured, with the apparatus available, to an accuracy within 5 per cent. Electrode areas of 50 to 500 sq. cm. (7.7 to 77.5 sq. in.) should prove suitable. Because of possible catalytic or corrosive effects of some compounds on certain metals, the electrodes should be nickel, gold, or platinum plated. The insulating material used to support the electrodes shall be capable of withstanding the wide temperature range to which the cell is subjected and preferably shall be of an inorganic material such as a ceramic material or suitable glass. A test run on the empty cell shall be made over the temperature range chosen in order to determine whether any leakage is present. A conductivity cell that has been found suitable is described in Appendix III.

#### Preparation of Test Specimen

43. (a) A representative sample shall be taken from the original package, melted, and poured directly into the testing container. Care shall be exercised in melting and pouring the compound not to overheat nor to entrap air in it. A melting and pouring temperature of approximately 50 C. above the softening point (ball and shouldered ring method) as determined under Section 2 (b) is recommended. The quantity of the sample depends upon the capacity of the resistivity cell used, but in any case it shall be sufficient to permit three separate determinations. Before filling, the conductivity cell shall be heated to slightly above the pouring temperature of the compound.

(b) A suggested procedure in filling the cell, especially in the case of the

higher melting compounds, is to determine the quantity of compound necessary to just fill the cell with the electrodes in position. In the case of coaxial cones or concentric cylinders the proper quantity of the heated compound shall be first poured slowly into the outer cone or cylinder. Any bubbles which may form on the surface of the compound shall be removed by a quick application of a flame from a bunsen burner. Immediately the inner electrode shall be slowly lowered into the compound and a thermometer placed in the well.

#### Procedure

44. (a) The conductivity cell, after filling, shall be placed in an oil or air bath having suitable temperature control, and sufficient time allowed to elapse to bring the bath and cell to temperature equilibrium at each test temperature. The temperature of the cell shall be determined by two mercury thermometers placed in contact with the electrodes. The temperature of the bath shall be determined by a mercury thermometer placed near the cell. The temperature of the bath shall be within 1 C. of the test temperature when readings are taken. The temperatures of the cell thermometers shall agree within 0.5 C. of each other and within 1 C. of the bath temperature when readings are taken.

(b) The volume resistance shall be measured at each temperature in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257) of the American Society for Testing Materials,<sup>4</sup> with particular attention to Sections 7, 8, 9, 10, 15, and 17 for liquid insulating materials.

#### Report

45. The report shall include the following:

- (a) The type of conductivity cell used,
- (b) The distance between guarded and unguarded electrodes,
- (c) The area of guarded electrode,
- (d) The battery voltage, and
- (e) A curve plotted from the readings obtained over the temperature range chosen showing the relation of volume resistance in ohm-centimeter units and the temperature in degrees Centigrade.

NOTE.—In lieu of the information required in Paragraphs (b) and (c), a statement of the approximate over-all accuracy may be made.

#### POWER FACTOR AND DIELECTRIC CONSTANT

##### Purpose

46. The purpose of this test is to determine the power factor and the dielectric constant of solid filling and treating compounds at specified temperatures corresponding to those temperatures to which the materials are to be subjected in service.

##### Temperature Range

47. Since the power factor and dielectric constant of this class of materials vary with the temperature, a sufficient number of tests shall be made at different temperatures to establish the dielectric constant - temperature curve and the power factor - temperature curve over the range of temperature to which the respective material is subjected in service. Determinations shall be made at not less than three temperatures; a temperature near the lowest to which the material is to be subjected in service, ordinary room temperature of 20 to 25 C., and a temperature near the maximum temperature to which it is subjected in service.

NOTE.—For many materials the power factor—temperature curve may have a maxi-

mum, minimum, or other irregularity within the working range of temperatures. With such a material many determinations may be required to establish the curve.

### Test Specimens

48. The materials to which these tests are applicable fall into two classes as regards the most suitable type of specimen to be used in the measurement of power factor and dielectric constant. One class includes waxes and other materials which can be cast or pressed into sheet form and then measured between electrodes such as are used in making tests on porcelain, molded materials, and sheet materials. (See the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.)<sup>4</sup> The other class includes materials that are fluid at ordinary temperatures or can be made fluid by increasing their temperatures. Materials in the fluid state can be poured into a measuring cell such as is described in Section 42 and the determinations made from which the power factor and the dielectric constant can be calculated.

### Apparatus

49. In the measuring cell the distance between the electrodes and the area of the electrodes shall be such that a capacitance of not less than 100  $\mu\mu f$  will be obtainable. The voltage gradient shall not be over 5 v. per mil. Due to the catalytic or corrosive effects of some compounds on certain metals, the electrodes may need to be nickel, gold, or platinum plated. The insulating material used to support the electrodes shall be capable of withstanding the wide range of temperature to which the cell or material is to be subjected during the tests without any appreciable

effect upon the determinations being made. Certain ceramic materials, special glasses, and quartz have been found suitable as insulating supports for the electrodes between which the material is to be tested. One cell that has been found satisfactory for these determinations is described in Appendix III. Cells of different construction from the one described have been used with equally satisfactory results.

### Preparation of Test Specimen

50. A representative sample shall be taken from the original package, melted if necessary, and then either cast in the form of a disk or poured directly into the measuring cell. The use of the cell or the disk will depend upon the particular material. In heating and pouring the compound, care shall be exercised not to entrap air in the compound between the electrodes. A melting and pouring temperature of approximately 50 C. above the softening point (ball and shouldered ring method) as determined under Section 2 (b) is recommended. A sufficient quantity of the material shall be taken as the sample to enable making at least three determinations. Before pouring the compound into the container or cell, the container or cell shall be heated to a temperature slightly higher than the temperature at which the compound is being poured.

### Temperature Bath

51. The test specimen after preparation shall be placed in an oil or air bath having suitable temperature control, and sufficient time shall be allowed to elapse to bring the bath and specimen to the desired temperature equilibrium at each test temperature. The temperature of the electrodes and the compound shall be determined by two mercury thermometers, or two thermocouples with cold junctions placed in contact with the electrodes. The temperature



of the bath shall be determined by a thermometer, or a thermocouple with cold junction placed near the cell or the specimen. The temperature of the bath shall be within 1 C. of the desired test temperature when the determinations are made. The temperature of the specimen thermometers or thermocouples shall agree within 0.5 C. of each other and within 1 C. of the bath temperature when the readings are taken.

### Procedure

52. The power factor and dielectric constant shall be determined at the specified temperatures and frequencies in accordance with the methods outlined for 25 to 60 cycles, 1000 cycles, and 100 to 1500 kilocycles as described in the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S. T.M. Designation: D 150) of the American Society for Testing Materials.<sup>4</sup> Measurements shall be made at the frequencies to which the material will be subjected in service unless otherwise specified. When using a cell in making power factor and dielectric constant determinations, measurements shall be made at the temperature desired, first with air as dielectric and then with the compound in the cell as dielectric.

### Calculations

53. The power factor shall be calculated in accordance with the Tentative

Methods D 150, and the dielectric constant shall be determined for any given compound from the ratio of the capacitance of the cell with the compound as dielectric to the capacitance of the cell with air as dielectric. When the specimen under determination is in the form of a disk the air capacitance shall be calculated from the dimensions of the electrodes and the thickness of the dielectric. In this case, correction shall be made as outlined in the Tentative Methods D 150. When the sample has been poured between electrodes being held apart by separators, the air capacitance can be measured directly the same as with the cell, or it may be calculated from the dimensions of the electrodes and the thickness of the separators, taking into account the edge correction.

### Report

54. The report shall include the following:

- (a) The type of cell used and the dimensions of the cell,
- (b) The method used and the frequency at which measurements were made,
- (c) The voltage across the specimen during the test,
- (d) The dielectric constant, the power factor, and the respective temperatures at which the determinations were made, and
- (e) A statement of the approximate accuracy of the determinations.

## APPENDIX I

## CELL FOR DETERMINING COEFFICIENT OF EXPANSION

Figure 1 shows the metallic cell for coefficient of expansion determinations of solid filling and treating compounds. The cell consists of four principal parts: A steel cylinder, a metallic gasket, a steel cover, and a dummy or auxiliary cover for filling. The gasket must be of a metal which does not amalgamate with mercury.

The cylinder is about 2.5 in. (6.35 cm.) in internal diameter, and approximately 3 in. (7.6 cm.) in internal depth. The top of the cylinder is threaded to receive the steel cover and has a machined shoulder to seat a 0.003 in. thick metallic gasket. The cylinder may be of

one piece construction or fitted with a cap at the bottom similar to the top end.

The steel cover is carefully rounded on the under side to avoid air pockets. It is threaded into the top of the cylinder and seats on the metallic gasket. The center of the cover is threaded to receive a steel capillary tube of 0.018 in. in internal diameter.

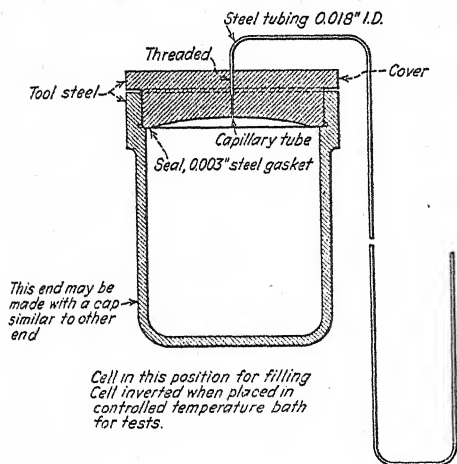


FIG. 1.—Metallic Cell for Coefficient of Expansion Determinations.

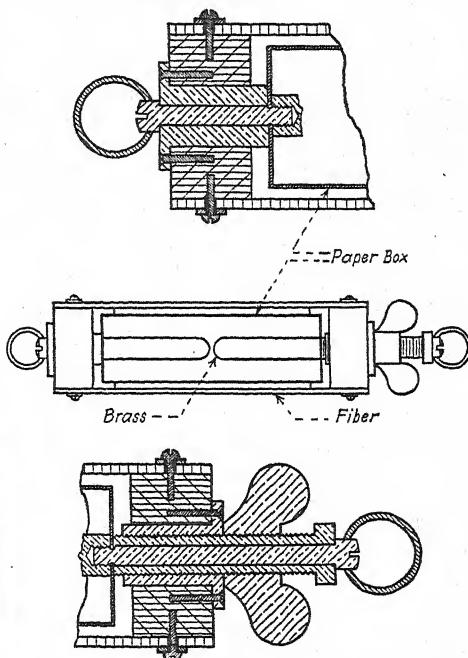


FIG. 2.—Container for Dielectric Strength Test Showing Electrodes in Place.

## APPENDIX II

## DEVICE FOR DETERMINING DIELECTRIC STRENGTH

Because of the great difficulty in removing most solid filling and treating compounds from the container, it is desirable to use a test assembly having an inexpensive container which can be thrown away after a test. A device of this sort is illustrated in Fig. 2.

The test assembly consists of a framework made from laminated phenol fiber, large enough to hold loosely a box of heavy paper or pasteboard 10 by  $1\frac{3}{4}$  by  $1\frac{3}{4}$  in. in inside dimensions, with brass bushings centrally inserted in each end piece to hold the electrode rods. The

electrodes, which are separable by means of screw joints, are inserted through small holes in the ends of the paper box and clamped to make a compound-tight joint. For the electrodes, a metal is selected that will give minimum gap changes with temperature. Steel has been found quite satisfactory. The proper

electrode spacing may be obtained by means of an adjusting screw on the right-hand end.

After use, the electrode supporting screws are backed off; the pasteboard container, holding part of the electrodes, is then easily withdrawn. The electrode parts are salvaged by melting the compound, and the used pasteboard container discarded.

### APPENDIX III

#### MEASURING CELL FOR RESISTIVITY, POWER FACTOR, AND DIELECTRIC CONSTANT MEASUREMENTS

A conductivity cell that has been developed for the purpose of measuring the volume resistance as well as other electrical constants of solid

was chosen after experimenting with various types for the following points of simplicity and efficiency:

1. Ease of insulating electrodes.

2. Large area of electrodes in compact form, the outer one of which also serves as container for the compound. This outer electrode is also exposed directly to the heating medium and aids in rapid changes from one temperature to another and also promotes uniform temperature distribution to the comparatively thin layer of compound in contact with it.

3. Comparative ease in assembling, disassembling, and cleaning the cell.

This type of cell permits the use of a very small amount of insulating material to insulate the electrodes. Mycalex has been found to be a satisfactory material for this purpose, due to the fact that it can be machined to size and has good insulation resistance characteristics over the temperature range at which these compounds are normally tested. Also, it apparently is unaffected by the solvents used in the cleaning operation.

A suggested method of cleaning the cell is to remove the bottom retaining ring and to hang the cell in an oven by a hook fastened through a hole drilled in the protruding stem. Upon heating, the outer cylinder will slide away from the inner electrode and both parts will drain fairly clean of the compound. In order to speed up this cleaning, the flame of a bunsen burner may be applied directly to the cell after removing the cap and suspending the cell by the protruding stem. Further application of the flame to the cylinder and inner electrode after they separate will rapidly remove most of the compound and the final cleaning of each part can be accomplished by suitable solvents.

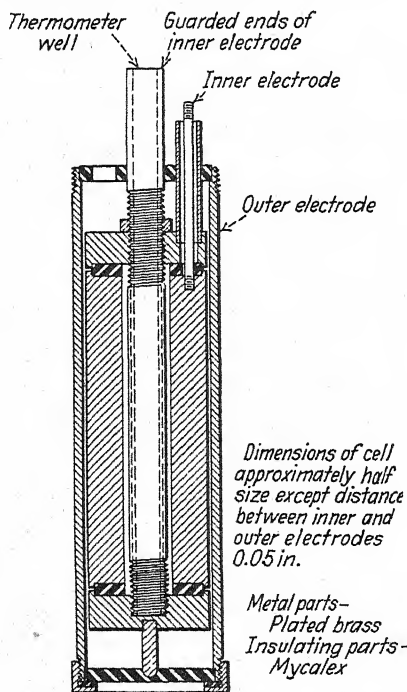


FIG. 3.—Measuring Cell for Resistivity, Power Factor, and Dielectric Constant Determinations.

filling and treating compounds and oil is shown in Fig. 3. The concentric cylinder type of cell

# Standard Method of Test for ARC RESISTANCE OF SOLID ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 495 - 42

ADOPTED, 1941; REVISED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 495; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method of test is intended for determining the relative arc resistance of all solid electrical insulating materials. It serves to classify materials broadly rather than to bring out slight differences between materials of similar composition. For at least the first three steps of the test, when the testing current is only 10 milliamperes, differences must be in the order of 2 to 1 (in seconds) in order to be significant. The general grouping of several widely used classes of insulating materials is approximately as follows:

Hot molded or laminated materials (general).....	30 sec. and under
Hot molded or laminated materials (higher arc resistant types).....	60 to 180 sec.
Cold molded materials (nonrefractory).....	60 to 300 sec.
Cold molded materials (refractory).....	240 to 480 sec.
Special materials (porcelain, wet or dry process; fused quartz; best grade slate; and similar materials).....	420 to 660 sec.

## Nature of Test

2. A high-voltage low-current a-c. arc, drawn between two tungsten pointed electrodes resting upon the surface of

the material under test, is increased at 1-min. intervals until the material fails by becoming conducting. The time required to reach failure is a measure of the arc resistance of the material.

## Apparatus

3. The apparatus shall consist of the following:

(a) *Transformer*.—A transformer of any convenient rating equal to or greater than 12 kv. and 1.2 kva. The source of supply of a-c. energy shall be not less than 1 kva., and the frequency shall be 60 cycles per sec. (Note 1).

NOTE 1.—When it is desired to test only organic insulating materials, a transformer rating of 12 kv. and 0.5 kva., or less, depending upon the material, will be satisfactory and the higher current steps will not be required.

(b) *Ammeter*.—A suitable milliammeter (Note 2) for measuring the secondary current of the transformer. The secondary current shall be controlled in magnitude by a variable resistance or inductance in the primary circuit.

NOTE 2.—Depending upon the transformer design, more or less high-frequency current is

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as standard from 1938 to 1941, being revised in 1941.

present in the high-voltage circuit, and unless proper precautions are taken, erroneous milliammeter readings may be obtained.

(c) *Interrupter*.—A motor-driven interrupter to give the required cycles for the three lower steps of the test. A wiring diagram of the equipment required for the test is shown in Fig. 1.

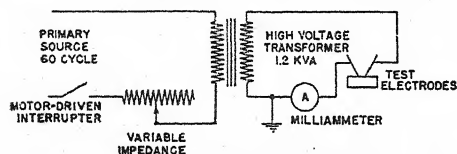


FIG. 1.—Wiring Diagram.

(d) *Electrodes*.—Electrodes consisting of threaded steel rods 0.186 in. in diameter, equipped with tungsten wire tips 0.060 in. in diameter, which shall have a conical point with a 60-deg. included angle. The electrodes shall be screwed into brass inserts in a wooden block at 45 deg. to the vertical and shall be adjusted to give a gap of 0.320 in. (0.813 cm.). Both electrodes shall lie in the same vertical plane. The weight of the block shall be supported on the two electrode points and on a third adjustable screw at the back of the block. The method of mounting the electrodes is illustrated in Fig. 2.

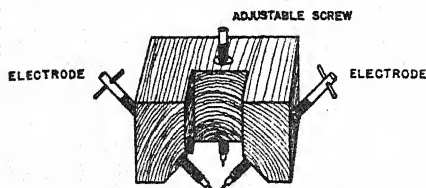


FIG. 2.—Electrode Mounting.

### Test Specimens

4. The test specimens may be of any size or shape, provided a flat, or nearly flat, surface of about 0.16 sq. in. (1 sq. cm.) in area and at least  $\frac{1}{8}$  in. in thickness, either one piece or in a pile-up of thin sheets, is available for each test.

### Procedure

5. (a) The arc-resistance test shall be made in air, sheltered from drafts, and with the specimen at room temperature. The electrode points shall be kept sharp and shall rest on the specimen in a horizontal position, as shown in Fig. 2. The open circuit voltage of the testing transformer shall be adjusted to from 12 to 15 kv. before starting the test.

(b) The sequence of steps, as shown in Table I, may be manually controlled by the operator or automatically controlled by a timing and switching circuit. The time in seconds from the start of the test until the failure of the material shall be recorded. Failure occurs when the surface of the material under test

TABLE I.—SEQUENCE OF 1-MIN. CURRENT STEPS.

Current, ma.	Time Cycle <sup>a</sup>	Approximate Rate of Heat Generation, watts	Total Time, sec.
10.....	$\frac{1}{4}$ sec. on, $1\frac{3}{4}$ sec. off	3	60
10.....	$\frac{1}{4}$ sec. on, $\frac{3}{4}$ sec. off	6	120
10.....	$\frac{1}{4}$ sec. on, $\frac{1}{4}$ sec. off	12	180
10.....	continuous	24	240
20.....	continuous	34	300
30.....	continuous	45	360
40.....	continuous	56	420
50.....	continuous	67	480
60.....	continuous	79	540
70.....	continuous	91	600
80.....	continuous	103	660
90.....	continuous	115	720
100.....	continuous	128	780

<sup>a</sup> In the earlier steps an interrupted arc is used to obtain a less severe condition than the continuous arc; a current of less than 10 ma. produces an unsteady arc.

becomes conducting, at which time the arc in air disappears and a bright line appears between the electrodes (Note 3).

NOTE 3.—It is frequently difficult to determine the time of failure of arc-resistant materials, such as refractory materials, porcelain, etc., as the entire surface under test is at white heat for some time before failure. The results of these tests are therefore liable to be considerably influenced by the judgment of the operator. For materials which last through the 100-ma. step, the test shall be discontinued as such high currents rapidly burn away the electrodes and it is very difficult to determine the time of failure.

**Number of Tests**

6. Unless otherwise specified, ten tests shall be made.

**Report**

7. Unless otherwise specified, the report shall include the following:

(1) A suitable description of the material tested and the approximate thickness at the test location,

(2) The time in seconds for each failure and the average time of failure for the ten tests,

(3) The current step at which each failure takes place, and

(4) A general statement describing the behavior of the material during the test and the manner in which it fails (carbonizes, volatilizes, fuses, melts, or similar reaction).

# *Standard Methods of Test for*

## DIELECTRIC STRENGTH OF ELECTRICAL INSULATING MATERIALS AT COMMERCIAL POWER FREQUENCIES<sup>1</sup>



A.S.T.M. Designation: D 149 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 149; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods of test outline procedures for the determination of dielectric strength of solid, semisolid, and liquid electrical insulating materials, except where a specific material requires a certain amount of special treatment. In such cases, reference should also be made to the applicable A.S.T.M. methods for the material to be tested (Note).

NOTE.—The following methods and specifications of the American Society for Testing Materials now provide for dielectric strength tests:

Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48),<sup>3</sup>

Tentative Methods of Testing Varnishes Used for Electrical Insulation (A.S.T.M. Designation: D 115),<sup>3</sup>

Standard Methods of Testing Electrical Porcelain (A.S.T.M. Designation: D 116),<sup>3</sup>

Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117 - 43),<sup>3</sup>

Tentative Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117 - 46 T),<sup>3</sup>

Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (A.S.T.M. Designation: D 176),<sup>3</sup>

Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (A.S.T.M. Designation: D 202),<sup>3</sup>

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229),<sup>3</sup>

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation D 295),<sup>3</sup>

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 348),<sup>3</sup>

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (A.S.T.M. Designation: D 349),<sup>3</sup>

Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350),<sup>3</sup>

Standard Methods of Testing Pasted Mica Used in Electrical Insulation (A.S.T.M. Designation: D 352),<sup>3</sup>

Standard Methods of Test for Product Uniformity of Phenolic Laminated Sheets (A.S.T.M. Designation: D 634),<sup>3</sup>

Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 902),<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1922 to 1944, being revised in 1923, 1924, 1926, 1927, 1930, 1931, 1932, 1933, 1934, 1936, 1938, 1939, and 1940.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for Friction Tape for General Use for Electrical Purposes (A.S.T.M. Designation: D 69),<sup>3</sup> and Tentative Specifications for Rubber Insulating Tape (A.S.T.M. Designation: D 119).<sup>3</sup>

### Nature and Types of Tests

2. (a) The dielectric strength of an insulating material is the maximum potential gradient that the material can withstand without rupture. It is calculated from the breakdown voltage and the thickness of the specimen at or near the point of rupture, and is commonly expressed in volts per mil or volts per millimeter. The value obtained in a test will depend upon the method and conditions of test.

(b) In general, the dielectric strength of insulating materials decreases with time of exposure to the electrical stress. Tests involving short exposures are, therefore, primarily comparative and not indicative of the breakdown of the materials under prolonged exposure to lower stresses.

(c) For quick determinations of dielectric strength, the short-time test has been devised. For fairly rapid determinations, but laying more emphasis upon the time factor, the step-by-step test has been arranged. For tests involving longer time factors, reference should be made to the applicable A.S.T.M. methods for the material to be tested (see Note, Section 1).

### Electrical Apparatus

3. (a) *Transformer*.—The desired test voltage may be most readily obtained by a step-up transformer energized from a variable low-voltage source. The transformer and its controlling equipment shall be of such size and design that, with the test specimen in circuit, the crest factor (ratio of maximum to mean effective) of the test voltage shall not differ by more than plus or minus 5 per cent from that of a sinusoidal wave

over the upper half of the range of test voltage. The crest factor may be checked by means of a sphere gap or peak-reading voltmeter in conjunction with a r.m.s. voltmeter. For test specimens of small capacitance, a testing transformer as small as 500-v.-amp. rating or a well-designed potential transformer may be adequate. For specimens of high capacitance, a transformer of ample kilovolt-ampere rating must be used. Where the wave-form cannot be determined conveniently, a transformer having a rating of not less than 2 kva. shall be used for voltages not exceeding 50,000 v., and one having a rating of not less than 5 kva. for voltages exceeding 50,000 v. Tests shall be made at commercial power frequencies.

NOTE.—When a transformer is used at voltages lower than its full rating, the current drawn from the high voltage winding should not exceed the full-load full-voltage current rating.

(b) *Circuit Breaker*.—The test transformer circuit shall be protected by an automatic circuit-breaking device designed to open instantaneously on the current produced by breakdown of the test specimen. Excessive flow of current at the time of breakdown causes pitting and heating of the electrodes, and thereby increases the work of electrode maintenance and time of testing.

(c) *Voltage Control*.—The rate of voltage rise shall not, for short-time tests, vary more than plus or minus 25 per cent from the specified rate. Control of voltage may be secured in one of several ways: (1) variable-ratio auto-transformer, (2) resistance-potential divider, (3) generator-field regulation, or (4) induction regulator. Preference should be given to equipment having an approximately straight-line voltage-time curve over the desired operating range. Motor drive, with variable speed control, should be preferred to manual



drive because of the difficulty in maintaining reasonably uniform rate of voltage rise with the latter.

NOTE.—A small variable auto-transformer, motor controlled, with automatic limit features, is described in the Appendix.

(d) *Voltmeter*.—The voltage shall be measured by an approved method which gives root-mean-square values, preferably by means of: (1) a voltmeter connected to the secondary of a separate potential transformer, (2) an electrostatic voltmeter in the high-tension circuit, or (3) a voltmeter connected to a well-designed tertiary coil in the test transformer. A voltmeter connected to the low-voltage side of the testing transformer may be used only if the ratio of transformation does not change appreciably with load.

(e) *Rate of Rise of Voltage*.—The rate of voltage rise may be calculated from measurements of time required to raise the voltage between two prescribed values. When motor-driven regulating equipment is used, the speed-control rheostat may be calibrated in terms of voltage rise for any particular test transformer.

### Electrodes

4. The dielectric strength of an insulating material varies with the thickness of the material and the area and geometry of the test electrodes. Tests made with different electrodes are not comparable. Where materials are made up into forms of uniform thickness, such as sheets and plates, tests shall be made upon that thickness of material. In other cases, a thickness of test specimen and diameter and shape of electrode have been selected that are compatible with convenience of testing. The electrodes used shall be as follows:

(a) *Thin Solid Materials* (Sheets and Plates):

(1) *Wide*.—Metal disks 2 in. in diameter, and 1 in. in length with the edges rounded to a radius of  $\frac{1}{4}$  in.

(2) *Narrow* (Tapes).—Opposing cylindrical rods  $\frac{1}{4}$  in. in diameter with edges rounded to a radius of  $\frac{3}{8}$  in. Upper movable electrode shall weigh  $0.1 \pm 0.005$  lb.

NOTE.—For rubber and friction tapes, reference should be made to the Tentative Specifications for Rubber Insulating Tape (A.S.T.M. Designation: D 119),<sup>3</sup> and for Friction Tape for General Use for Electrical Purposes (A.S.T.M. Designation: D 69)<sup>3</sup> of the American Society for Testing Materials.

(b) *Thick Solid Materials*.—Metal disks 1 in. in diameter, and 1 in. in length with edges rounded to a radius of  $\frac{1}{8}$  in.

(c) *Liquids*.—Metal disks 1 in. in diameter with square edges, mounted with axes horizontal.

(d) *Compounds*.—Hemispherical metal electrodes  $\frac{1}{2}$  in. in diameter.

(e) *Materials Requiring Special Treatment* (Tubes, Varnishes, etc.).—Reference shall be made to the specific methods for the materials to be tested (see Note, Section 1).

### Test Specimens

5. (a) The dielectric strength of an insulating material varies with the thickness of the test specimen. Tests on specimens of different thickness are therefore not comparable. The specimens shall be representative of the material to be tested. Sufficient material shall be available to permit making five satisfactory tests (Section 11).

(b) *Thin Solid Materials* (Sheets and Plates).—The test specimens need be only of sufficient area to prevent flash-over under the conditions of test.

(c) *Thick Solid Materials*.—The breakdown voltage of thick solid materials is so high that special test specimens, of reduced thickness, shall be cut

or molded. For details of these specimens, reference shall be made to the specific methods for the materials to be tested (see Note, Section 1).

(d) *Liquids*.—For testing liquids, it is necessary to use a special test cup with electrodes rigidly mounted with their axes in a horizontal plane. The cup shall be of such design that no part of it is less than  $\frac{1}{2}$  in. from any part of the test faces of the electrodes, when the latter are in testing position. It shall be made of a material of high dielectric strength, insoluble in, and unattacked by any of the test or cleaning liquids and nonabsorbent as far as moisture and the test and cleaning liquids are concerned. The top of the cup shall be about 33 mm. ( $1\frac{1}{4}$  in.) above the top of the electrodes.

(e) *Other Materials*.—For a description of the test specimens of other materials, reference shall be made to the specific methods for the materials to be tested (see Note, Section 1).

#### Conditioning of Test Specimens

6. (a) The dielectric strength of most insulating materials varies with temperature and humidity. Usually it is desirable to determine the dielectric behavior of a material over the range of temperature and humidity to which it is likely to be subjected in use. As this varies for different materials reference shall be made for information concerning the conditioning treatment of a particular material to the specific method for that material (see Note, Section 1). Materials may be conditioned in a suitably controlled chamber. The test specimens shall be kept in the chamber long enough to reach a uniform temperature and humidity before voltage is applied. The dielectric strength tests shall be made on the specimen while still in the conditioning chamber. For purpose of tests, a high-voltage

conductor may be conveniently carried into the chamber through an insulating bushing.

(b) For tests made in air, use may be made of any well-designed oven of sufficient size to hold the test equipment. It should be provided with some means of circulating the air, so that approximately constant temperature is maintained around the test specimen, and with a thermometer or thermocouple for measuring the temperature as near the point of test as practicable.

(c) For tests under oil, use may be made of an oil bath, provided with some means for circulating the oil, so that the temperature is substantially uniform around the test specimens, and with a thermometer or thermocouple for measuring the temperature as near the point of test as practicable.

#### Surrounding Medium for Solid Materials

7. In general, it is preferable to test materials in the medium, whether air or oil, in which they are to be used. Where conditions of use are not well defined, materials should be tested in air up to the point where the breakdown is so high that an excessive amount of material is required to prevent flashover or excessive burning of the surface. For specimens having a high breakdown, such as the thicker and high-grade materials, it is usually necessary to make dielectric strength tests under oil, however, it should be understood that breakdown values obtained under oil are not comparable with those obtained in air. For the medium to be employed on a particular material, reference shall be made to the specific method for the material to be tested (see Note, Section 1).

#### Adjustment and Care of Electrodes

8. (a) Electrodes shall be held truly coaxial. Where electrodes have flat

test faces, the latter shall be parallel to each other. The test faces shall be kept smooth and polished and free from pitting.

(b) Liquid testers shall be cleaned at the beginning of each day's testing and after any breakdown occurring at a value below the acceptable unit for the liquid under test. The electrodes and the test cup shall be wiped clean with dry, calendered tissue paper or with a clean, dry chamois skin. In test cups provided with a micrometer screw adjustment, the spacing of the electrodes shall be adjusted to 0.100 in. by means of the micrometer. In test cups not so equipped, the spacing of the electrodes of the test cup shall be checked with a standard round gage having a diameter of 0.100 in. or flat steel "go" and "no-go" gages having thicknesses of 0.0995 and 0.1005 in., respectively, and the electrodes locked in position. It is important to avoid touching the electrodes or the cleaned gage with the fingers or with portions of the tissue paper or chamois skin which have been in contact with the hands. The electrodes and cup shall then be rinsed with dry lead-free gasoline, Stoddard solvent, or dry, water-white kerosine until they are entirely clean. Care shall be taken not to touch the electrodes or the inside of the cup after cleaning, so as to avoid any possible contamination. After thoroughly cleaning the test cup, it shall be filled with a sample of the cleaning fluid and voltage applied with uniform increase at the rate of approximately 3 kv. per sec. (rms. value) until breakdown occurs. If the breakdown is not less than the established value of the liquid being tested, the cup shall be considered in suitable condition for testing. If a lower value is obtained, the cup shall again be thoroughly cleaned and the test repeated. A cleaning fluid whose

breakdown is not less than the established value of the liquid being tested must be used. Evaporation of the cleaning fluid from the electrodes may chill them sufficiently to cause moisture to condense on their surfaces. For this reason, after the final rinsing with cleaning fluid, the cup must immediately be flushed with the liquid to be tested, following which the cup should be filled for the test.

#### Preparation of Test Specimen

9. (a) *Solid Materials*.—In the preparation of test specimens from solid materials, care shall be taken to have the surfaces adjacent to the electrodes parallel and as plane and smooth as the material permits.

(b) *Liquids*.—The dielectric strength of liquid dielectrics may be markedly altered by the migration of impurities through the liquid. In order that representative test specimens may be obtained, the container shall be gently tilted or inverted several times in such a way that any impurities present will be thoroughly mixed with the dielectric. Too rapid agitation is undesirable, as it introduces an excessive amount of air into the mixture. Immediately after mixing, the test cup shall be filled with liquid to a height of not less than 20 mm. (0.787 in.) above the top of the electrodes. In order that entrapped air may escape, the container shall be gently rocked a few times and the liquid allowed to stand in the cup for 3 min. before voltage is applied. Where more than one test is made on the same specimen, the liquid shall be allowed to stand for 1 min. between tests.

(c) *Other Materials*.—For the preparation of specimens of insulating compounds, varnish films, etc., reference shall be made to the specific methods for the materials to be tested (see Note, Section 1).

### Application of Voltage

10. (a) *Short-Time Test*.—The voltage shall be increased from zero to breakdown at a uniform rate. For solid materials, the rate of rise shall be 0.5 or 1.0 kv. per sec., depending on the total test time required and the voltage-time characteristic of the material. For the rate applicable to a given material, reference shall be made to the test method for that material (see Note, Section 1). For liquid materials, the rate of rise shall be 3 kv. per sec.

(b) *Step-by-Step Test*.—An initial voltage shall be applied equal to 50 per cent of the breakdown voltage in the short-time test, adjusted as shown in the following table:

Breakdown Voltage by Short-Time Method	Adjust 50 per cent of Breakdown Voltage to the Nearest
25 kv. or less.....	1.0 kv. (except as otherwise specified)
Over 25 to 50 kv., incl. ....	2.0 kv.
Over 50 to 100 kv., incl. ...	5.0 kv.
Over 100 kv. ....	10.0 kv.

The voltage shall then be increased in equal increments as stated in the various material specifications, the voltage being held at each step for a definite time as stated in the specifications. The change from each step to the next higher shall be made as rapidly as possible, and the time of change included in the succeeding test interval.

### Number of Tests

11. Unless otherwise specified, five tests shall be made. If the average deviation from the mean exceeds 10 per cent, or if any individual test deviates

more than 15 per cent from the mean, five additional tests shall be made. The dielectric strength shall be determined from the average of all tests.

### Thickness of Solid Materials

12. Unless otherwise specified, the number of thickness determinations shall be at least equal to the number of punctures. The measurements shall be made on material undisturbed by breakdown, but as near the point of breakdown as possible. Measurements shall be made in accordance with the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

### Report

13. Unless otherwise specified, the report shall include the following:

- (a) The thickness of the specimen as determined in accordance with Section 12,
- (b) Total volts at each puncture,
- (c) Volts per mil for each puncture,
- (d) The average, maximum, and minimum volts per mil for each sample,
- (e) The temperature of the test specimen,
- (f) The percentage relative humidity of the surrounding air,
- (g) The conditioning treatment,
- (h) The duration of the test,
- (i) In the step-by-step test, the value of the initially applied voltage and the value of the voltage at each step, and
- (j) The size and type of electrodes.

(See Appendix, p. 130)

## APPENDIX

A simple form of motor-operated voltage controller is shown in Fig. 1. The control element is a variable auto-transformer, *A*, of the slide-wire type, the slider of which is driven through a system of reduction gearing, *B*, by a small direct-current motor, *C*. The auto-transformer supplies the primary of the testing transformer.

rise. Rapid adjustment of the rate of rise to the value desired is thereby secured without subsequent measurements. In reverse, the resistor is out of circuit, so that the motor operates at maximum speed.

Over-travel of the motor is prevented by connecting the motor operating circuits through segments, *EF*, on the insulating wheel, *D*, which

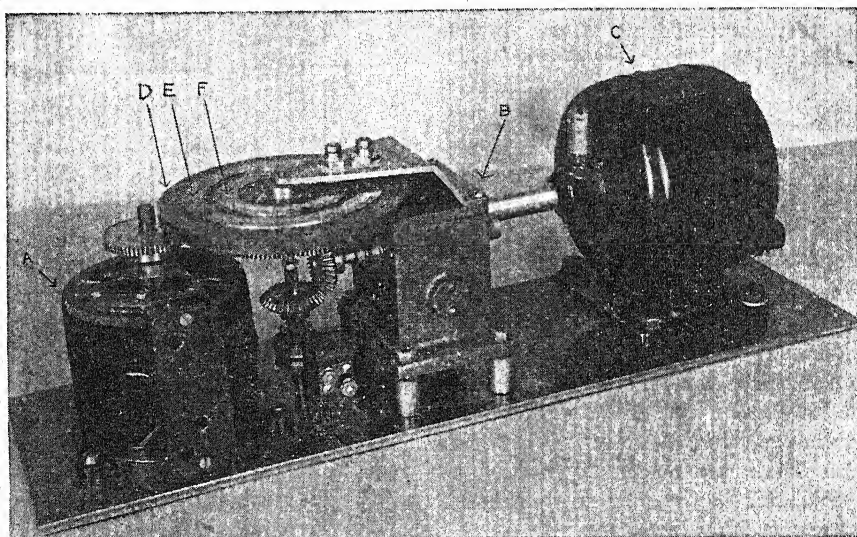


FIG. 1.—Variable Auto-Transformer, Motor Controlled, with Automatic Limit Features.

The motor is provided with a reversing switch, so that it may be operated either forward or in reverse. For forward operation, a variable resistor is connected in series with the armature so that the speed may be adjusted to any required value. Where one specific testing transformer is used, the control handle of the resistor may be calibrated in terms of rate of voltage

is attached to one of the gears. One segment is connected in the forward circuit, and the length of this is adjusted so that the circuit is automatically opened when the maximum available voltage is reached. The other segment is connected in the reverse circuit, and this is timed so that the circuit automatically opens when the voltage is reduced to zero.

# Standard Methods of Test for CONDUCTING PATHS IN ELECTRICAL SLATE<sup>1</sup>



A.S.T.M. Designation: D 273 - 40

ADOPTED, 1940.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 273; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods include two procedures for testing electrical slate for conducting paths or veins. The general fields of application and limitations of the two procedures are described in the sections on application which precede each of the methods. Both methods are nondestructive and may thus be used for routine factory or quarry inspection work.

## METHOD A

### Application of Method A

2. This method of test employs high voltage at very high frequencies obtained from a simple spark coil. Numerical readings are not obtainable with this test procedure, but it can be depended on to locate conducting paths or veins with considerable certainty and will, furthermore, give a rough, comparative indication of the impedance of the slate. It is recommended pri-

marily as a routine factory or quarry test, and since the apparatus required is very simple and inexpensive, it is applicable even where a comparatively small quantity of slate is to be tested. If the spark coil employed is of ordinary size, this method is much less hazardous to the operator than any method (such as Method B) which employs high voltage at commercial frequencies.

## Apparatus

3. The apparatus shall consist of the following:

(a) *Spark Coil*<sup>3</sup>—A spark coil of the vibrator type capable of giving a spark from  $\frac{3}{8}$  to 1 in. in length between needle points.

(b) *Current Source*.—Six volts direct current.

NOTE 1.—An automobile-type storage battery of about 100-amp-hr. capacity is suggested.

(c) *Electrodes*.—One electrode consisting of a sharp-pointed No. 8 B. & S. gage bare copper wire, about 4 in. in length, mounted on the end of a hard

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1927 to 1940, being revised in 1936.

<sup>3</sup> A Model T Ford ignition-type spark coil has been found satisfactory.

rubber rod about 1 in. in diameter and 1 ft. in length, or a multiple-point comb type electrode mounted on a similar hard rubber rod as shown in Fig. 1. The comb type electrode permits the exploration of large areas more rapidly than the single-point electrode. The electrode used shall be connected to the high-tension side of the spark coil with high-grade automobile ignition wire, or equivalent, not more than 10 ft. in length. The other electrode shall be a sheet of metal of suitable size on which the slate under test can be laid. This shall be connected to the

### Conditioning

6. No conditioning of the reference standard or the test specimen is ordinarily required, if the surfaces appear dry. As surface moisture tends to increase the brush discharge, wet specimens shall be dried in an oven at 150 F. (65 C.) for at least 16 hr. before testing.

### Procedure

7. (a) With both electrodes connected to the spark coil and while supporting the pointed ungrounded electrode by the insulating handle, the spark coil vibrator shall be adjusted to give as smooth and

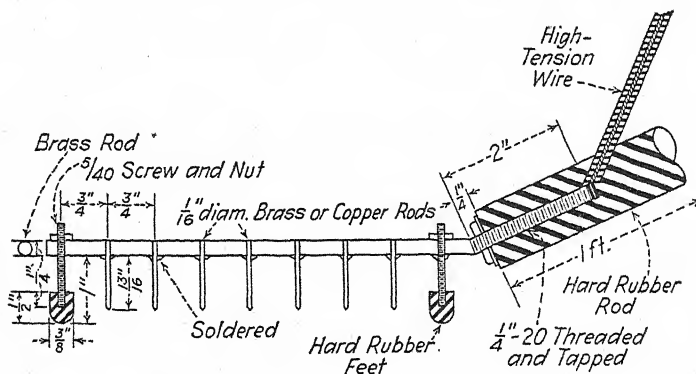


FIG. 1.—Suggested Multiple-Point Electrode for Exploring Slate for Conducting Paths and Veins by the Spark Coil Method.

other terminal of the spark coil which shall be grounded.

### Test Specimen

4. The test specimens may be of any size and shall have all surfaces, including edges, sand-rubbed or hone-finished.

### Reference Standard

5. A reference standard consisting of a sheet of electrical slate of a quality to be agreed upon between the manufacturer and the purchaser shall be used. This standard may be of any convenient size such as  $\frac{1}{2}$  by 12 by 12 in.

steady a tone as possible. The pointed ungrounded electrode shall then be brought within about  $\frac{3}{8}$  to 1 in. (depending upon the capacity of the coil used) of the plate electrode, and a continuous spark discharge should take place.

(b) The reference standard and the test specimen shall be laid side by side on the grounded electrode and the pointed electrode brought to within about  $\frac{1}{4}$  in. of the face of the standard. Under this condition a small amount of typical bluish brush discharge will be noted at the point. The test specimen shall then be carefully explored over its entire



face and edges with the pointed electrode, holding it about  $\frac{1}{4}$  in. from the surfaces of the specimen. In the case of the comb type electrode, the correct spacing between the points and the surface is definitely fixed. When a conducting vein or path is located, sparks will jump and scatter to it in lightning-like forks over the surface of the specimen (Note 2). Slate that shows conducting paths or veins under this test is unsatisfactory for electrical purposes and should be rejected.

NOTE 2.—The intensity of the brush discharge depends in general on the impedance of the slate, that is, the lower the impedance the greater the brush discharge. Unless the slate is to be used in apparatus where practically any leakage is objectionable, slight increase in the brush discharge noted on the specimen as compared with that on the standard is permissible, provided, however, that it is essentially uniform over the entire area of the specimen. Such an increase in the brush discharge must not be confused with the sparks which occur when an actual conducting vein is approached. Once observed, this latter sparking is unmistakable as it will be of marked intensity both visibly and audibly and will tend to branch out in many directions. The veins are sometimes confined to narrow ribbon-like boundaries that run the full length of the specimen. Others are more generally distributed and in these cases the spark discharge sometimes will enter the surface at one point, emerge at another and re-enter again at still another indicating the presence of substrata conducting paths.

## METHOD B

### Application of Method B

8. This method of test is primarily recommended for use when numerical readings are considered necessary. Fundamentally, this procedure measures the leakage current when the slate is subjected to a high voltage of commercial frequency. It requires a moderate outlay for apparatus and unless surrounded by proper safeguards should be used only by an operator experienced in working with high voltages. It is

thus primarily a laboratory method but is applicable to routine factory or quarry inspection service where the volume of slate to be tested is sufficiently great to justify the expense of the apparatus and of providing suitable safeguards.

### Apparatus

9. The apparatus shall consist of the following:

(a) *Transformer*.—A testing transformer rated at 1 kva. or larger, high-voltage winding for 10,000 v., provided with a middle tap.

(b) *Ammeters*.—Two similar alternating-current ammeters having a range of 200 or 250 ma.

(c) *Current Source*.—A source of alternating current having a frequency of 60 cycles per second, of approximately sine-wave shape, and of a voltage suited to the primary winding of the testing transformer.

(d) *Electrodes*.—Electrodes consisting of two metallic disks each having a contact area of not less than 2 sq. in., mounted in insulating handles for the effective protection of the operator, and a plate electrode as wide and of the same length as the specimen to be tested. If the plate electrode cannot be conveniently made as long as the specimen, then the specimen shall be tested in sections until its entire area has been covered (see Note 4, Section 13).

### Test Specimen

10. The test specimens may be of any size, and shall have all surfaces, including edges, sand-rubbed or hone-finished.

### Conditioning

11. No conditioning of the test specimen is ordinarily required, if the surfaces appear dry. As surface moisture tends to increase the leakage current, wet specimens shall be dried in an oven at



150 F. (65 C.) for at least 16 hr. before testing.

### Procedure

12. (a) The electrodes shall be connected with the ammeters, and these in turn with the high-voltage terminals of the testing transformer, by flexible cables provided with insulation suitable for continuous use at 10,000 v. The ammeters likewise shall be suitably insulated from ground for operation at 10,000 v. and shall be screened or guarded against accidental contact by the operator.

(b) The plate electrode shall be connected to the midpoint tap of the test transformer.

(c) The primary of the transformer shall be connected to the current source through a series resistor or reactor which will limit the high-tension current under short-circuit to not more than 0.25 amp. The applied voltage shall be adjusted until the open-circuit voltage of the high-tension winding is 10,000 v.

### Leakage Currents

13. The leakage currents of the test specimen as shown by the ammeters shall be noted for the following two tests:

(a) With the specimen lying on the plate electrode, both faces of the specimen shall be gone over with the disk electrodes held approximately, but not less than, 2 in. apart. Observation shall be made of the deflection of both ammeters. (Notes 3 and 4.)

(b) The specimen shall be stood on edge on the plate electrode and the two movable electrodes shall be moved over the edges of the specimen with attendant observation of both ammeters (Note 3).

NOTE 3.—It is necessary that observation be made of both ammeters since the presence of a conducting path may result in the deflection of either or both instruments, depending on the course which the path takes.

NOTE 4.—Where a considerable number of specimens are to be examined for conducting paths and the factory arrangements are such that these can be moved forward on some type of conveyor, it may be possible to examine the specimens in transit. To this end it will be necessary to provide two sliding or rolling upper electrodes. The lower metal plate electrode, 6 in. broad or more, shall be so placed that the specimen to be examined will pass over it. The two upper electrodes, of the sliding or rolling type, shall be spaced approximately, but not less than, 2 in. apart, and extend in parallel lines across the specimen at a suitable height and so placed with respect to the lower electrode as to make contact with the upper surface of the specimen in the region which is passing over the lower electrode.

*Standard Methods of Test for*  
INSULATION RESISTANCE OF ELECTRICAL INSULATING  
MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 257 - 46

ADOPTED, 1933; REVISED, 1938, 1945, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 257; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover the determination of insulation and volume resistance and approximate surface resistance of electrical insulating materials; also means of converting the latter two into terms of volume and surface resistivities.

### DEFINITIONS

#### Definitions

2. (a) *Volume Resistance*.—The volume resistance between two electrodes which are in contact with, or embedded in a specimen, is the ratio of the d-c. voltage applied to the electrodes, to that portion of the current between them that is distributed through the volume of the specimen.

(b) *Volume Resistivity*.—The volume resistivity of a material is the ratio of the potential gradient in volts per centimeter parallel to the current flow in the material, to the current density in amperes per square centimeter.

NOTE 1.—Volume resistivity of a material is numerically equal to the volume resistance when measured between two electrodes which cover opposite faces of a centimeter cube of the material.

(c) *Surface Resistance*.—The surface resistance between two electrodes which are on the surface of a specimen is the ratio of the d-c. voltage applied to the electrodes to that portion of the current between them which is in a thin layer of moisture or other semi-conducting material that may be deposited on the surface.

(d) *Surface Resistivity*.—The surface resistivity of a material is the ratio of the potential gradient in volts per unit distance parallel to the current flow along its surface, to the current in amperes per unit width of surface.

NOTE 2.—Surface resistivity of a material is numerically equal to the surface resistance between two electrodes forming opposite sides of a square. The size of the square is immaterial.

(e) *Insulation Resistance*.—The insulation resistance between two electrodes which are in contact with, or embedded in a specimen, is the ratio of the d-c. voltage across the electrodes to the

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1926 to 1933, being revised in 1929 and 1932.

total current between them. It is dependent upon both the volume and surface resistivities of the material as well as upon the configuration of the electrodes.

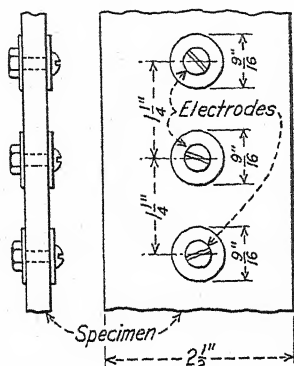
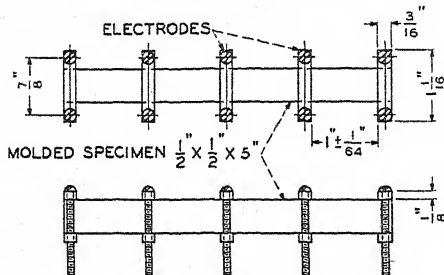


FIG. 1.—Application of Binding Post Electrodes to Flat, Solid Specimens.



NOTE.—The electrodes are either tin foil or other conducting material applied to both sides of the specimen. In the case of tin foil, it is wrapped around metal bars. After the bars have been mounted in position, the tin foil is pressed down with a thin tool along the edge of the bar to assure intimate contact with the insulation. The suggested dimensions have been found convenient.

FIG. 2.—Application of Strip Electrodes to Flat, Solid Specimens.

## TEST SPECIMENS AND TYPES OF ELECTRODES

### A. For Solid Insulating Materials

#### Test Specimen

3. The test specimen for solid insulating materials shall be either in the form of a flat plate, a cylinder, or a tube.

#### Electrodes

4. (a) It is important that good contact be made between the electrode and the insulating material. For repeated tests

at high relative humidities it is essential that the electrode metal be protected against tarnish.

(b) For insulation resistance determinations, the electrodes shown in Fig. 1 (binding posts), Fig. 2 (metal strips) or Fig. 3 (tapered pins) shall be used. For separating the insulation resistance into approximate surface resistance and volume resistance (from which approxi-

Holes Drilled  $\frac{3}{16}$ " in Diameter and Reamed to Fit Pratt and Whitney No. 3 Tapered Pins.

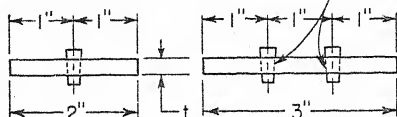


FIG. 3.—Application of Tapered Pin Electrodes.

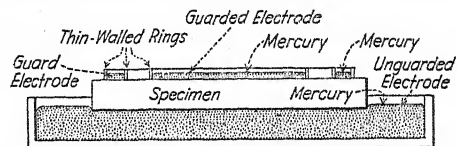


FIG. 4.—Application of Mercury Electrodes to Flat, Solid Specimens.

mate resistivities may be calculated) the guarded mercury electrodes (Fig. 4) are satisfactory though rather difficult to manipulate under abnormal atmospheric conditions.

(c) For insulation resistance measurements on molded or laminated materials the electrodes shown in Fig. 3 (tapered pins) shall be used, unless otherwise agreed upon by the manufacturer and the purchaser. Figs. 1 and 2 show electrodes which are in common use and which may be specified in place of the tapered pins, upon mutual agreement between the manufacturer and the purchaser.

#### Electrodes for Volume and Surface Resistance

5. When both the volume and sur-

face resistances are to be measured, there shall be applied to each specimen three electrodes designated, respectively, as the unguarded electrode, the guarded electrode, and the guard electrode (see Fig. 4). In the case of a flat specimen, the guard electrode shall be in the form of a ring surrounding the guarded electrode as shown in Fig. 4. In the case of tubular specimens the electrodes shall be applied as shown in Fig. 5.

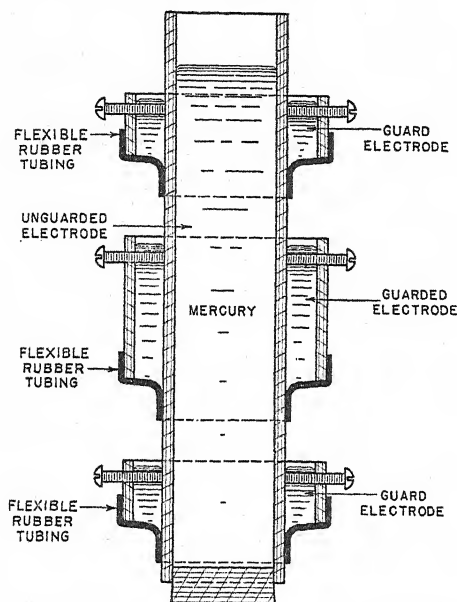


FIG. 5.—Application of Mercury Electrodes to Tubular Specimens.

NOTE.—In the case of a few nonabsorbent materials such as hard rubber and glass the insulation resistance measurement may be considered a measurement of the surface resistance from which the surface resistivity may be calculated. Such assumptions are undesirable, however, because they invariably lead to generalization at the expense of accuracy. Consequently, the plain insulation resistance test should be used except for research purposes.

#### B. For Liquid Insulating Materials

##### Sampling

6. Samples of oil shall be obtained as described in Sections 2 to 8 of the Stand-

ard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117) of the American Society for Testing Materials.<sup>3</sup> Samples of other liquid dielectrics shall be obtained in a similar manner with regard to cleanliness of containers and sampling apparatus and shall be representative of the lot to be tested. The quantity of the sample depends upon the type of resistivity cell used, but in any case it shall be sufficient to permit three separate resistivity determinations.

##### Electrodes

7. Suitable electrodes for liquid insulating material are parallel planes concentric cylinders, or coaxial cones. The distance between the electrodes shall be not less than 0.75 mm. (0.03 in.) nor more than 5 mm. (0.2 in.). The voltage gradient shall not exceed 1200 v. per mm. (30 v. per mil). The area of the electrodes shall be sufficiently large so that the current flow can be measured, with the apparatus available, to an accuracy of 5 per cent.

NOTE 1.—Electrode areas of 50 to 500 sq cm. (8 to 78 sq. in.) should prove suitable.

NOTE 2.—The electrode surfaces shall be made of a material capable of resisting attack by mild acids found in certain insulating oils such as petroleum oil, particularly after prolonged exposure to elevated temperatures. Metals which have been found satisfactory from this standpoint are gold, nickel, monel, and platinum. In general, plated surfaces have been found less satisfactory than solid metal electrodes.

NOTE 3.—Any of the cells described in Appendix III of the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150)<sup>3</sup>, are satisfactory for use in resistivity measurements.

NOTE 4.—If the shape of the electrodes is such that their effective area and the distance between them is difficult to measure, the volume resistivity may be calculated by the use of the following formula:

$$K = 36\pi \times 10^{11} \times C$$

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

where  $K$  = the constant by which the measured resistance is to be multiplied to give the volume resistivity, and

$C$  = the capacitance in farads of the electrode system with air as the dielectric. For methods of measurement of  $C$ , see the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup>

### Apparatus

8. (a) The apparatus shall consist of a source of direct-current potential, a galvanometer with suitable shunts, a calibrating resistance, reversing switches and keys. The apparatus shall conform to the requirements specified in the following Paragraphs (b) to (e).

(b) *Direct-Current Potential*.—A dry or storage battery may be used as a source of potential. A rectified alternating-current voltage supply may also be employed, if precautions are observed to maintain a sufficiently constant direct-current voltage.

(c) *Galvanometer*.—The galvanometer shall have as high a sensitivity as is consistent with reasonable stability of zero. A sensitivity of  $10^{-10}$  amp. per milliradian (1 mm. at 1-m. scale distance) is sufficient to measure resistance as high as  $10^{11}$  ohms with an accuracy of plus or minus 10 per cent. It should be well damped. The most convenient shunt is the type known as a universal or Ayrton shunt, whereby that fraction of the total current which passes through the galvanometer may be changed without changing the galvanometer damping.

(d) *Calibrating Resistance*.—The calibrating resistance shall be at least 100,000 ohms. One megohm is preferable for ordinary measurements.

(e) *Switches and Keys*.—All switches and keys shall be suitably insulated and guarded.

NOTE 1.—If resistances higher than  $10^{11}$  ohms are to be measured, a ballistic or a quadrant electrometer method is often employed. The latter method is described in the National Bureau of Standards *Scientific Paper No. 234*<sup>4</sup>. Other methods of measuring the minute currents flowing through these high resistances, employing vacuum tube electrometers,<sup>5</sup> are available and their use is permitted.

NOTE 2.—A bridge method of measuring insulation resistances up to  $10^{11}$  ohms may be used, if a high-resistance balance detector, such as a vacuum-tube voltmeter, is employed, and the necessary guarding requirements are observed.

### MEASUREMENTS

#### Measurements for Insulation Resistance

9. The following measurements shall be made when insulation resistance is to be determined:

(a) Dimensions of the electrodes (Figs. 1, 2 and 3),

(b) Distance between or spacing of the electrodes,

(c) The insulation resistance between the electrodes,

(d) The temperature of the material under test (see Section 11 (a)),

(e) The relative humidity of the surroundings (see Section 11 (a)), and

(f) Voltage used in measuring.

#### Measurements for Volume and Surface Resistance

10. The following measurements shall be made when both volume and surface resistance are to be determined by means of the specimens shown in Figs. 4 and 5:

(a) Area of the guarded electrode,

(b) Distance between the guarded electrode and the guard electrode,

(c) The over-all insulation resistance, that is, the resultant of the surface and

<sup>4</sup> H. L. Curtis, "Insulating Properties of Solid Dielectrics," National Bureau of Standards *Scientific Paper No. 234*, Vol. 11, p. 364.

<sup>5</sup> D. B. Penick, "Direct Current Amplifier Circuit for Use with the Electrometer Tube," *Review of Scientific Instruments*, Vol. VI, p. 113, April, 1935.

volume resistances, when the measurement is made with the guard electrode connected to the unguarded electrode,

(d) The volume resistance, which is the resistance between the guarded electrode and the unguarded electrode when the guard electrode is maintained at substantially the same potential as the guarded electrode,

(e) The temperature of the material under test (see Section 11 (a)),

(f) The relative humidity of the surroundings (see Section 11 (a)),

(g) Voltage used in measuring, and

(h) Thickness of the specimen.

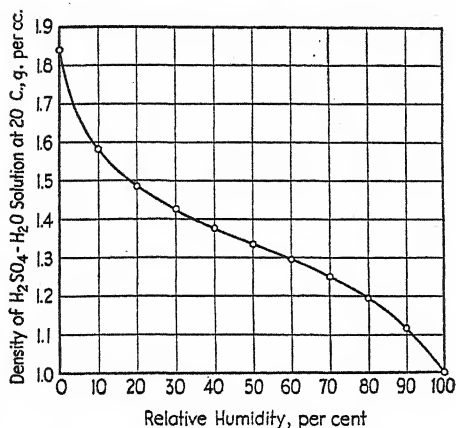


FIG. 6.—Relation of Relative Humidity of Air and Density of Acid-Water Mixture.

NOTE.—In the case of insulating oils it is often desirable to make measurements at an elevated temperature. To determine the suitability of oil for commercial apparatus tests are usually made at 85 C. or 100 C., or both.

## PROCEDURE

### Conditioning

11. (a) Unless otherwise agreed upon, determinations of insulation resistance of solid dielectrics shall be made on specimens which have been exposed for a period of 96 hr. in an atmosphere maintained at a relative humidity of  $90 \pm 2$  per cent and a temperature of  $35 \pm 1$  C.

( $95 \pm 2$  F.). When introduced into the conditioning chamber the specimen shall be at a temperature of 35 C. within  $+ 5$  or  $- 0$  C. Measurement of insulation resistance shall be made while the specimen is in the conditioning chamber maintained at the above specified relative humidity and temperature. An alternative conditioning procedure<sup>6</sup>, requires that the determination of insulation

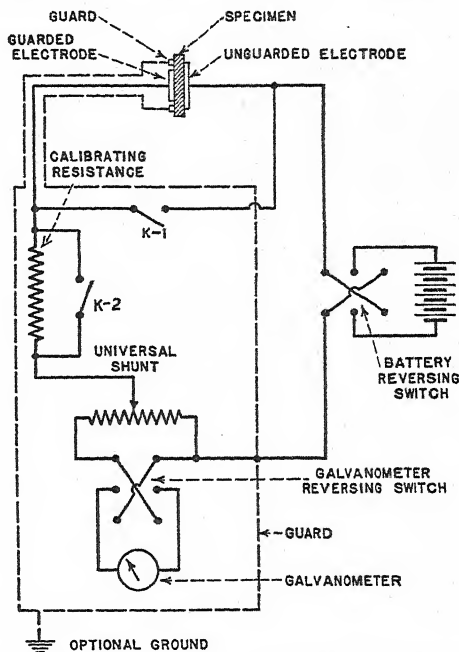


FIG. 7.—Diagram of Connections for Determination of Insulation Resistance.

resistance of solid dielectrics shall be made on specimens which have been immersed in water at 50 C. (122 F.) for a period of 96 hr., then cooled in water and wiped off. Other conditioning processes may be used if agreed upon by the manufacturer and the purchaser.

Where electrode leads pass through the walls of the chamber, care shall be

<sup>6</sup> This procedure is essentially the same as the conditioning procedure specified in the Joint Army-Navy Specifications for Plastic-Materials, Laminated Thermosetting Sheets and Plates (JAN-P-13), for Plastic-Materials, Molded Thermosetting (JAN-P-14), and for Plastic-Materials, Laminated, Thermosetting: Rods and Tubes (JAN-P-79).

taken to insure against current leakage at these points by suitable guarding or by passing leads through bushings of wax or other nonwetting materials.

(b) *Humidity and Temperature Control.*—Any method of humidity and temperature control may be used provided the stipulated limits of plus or minus 2 per cent for humidity and plus or minus 1 C. (2 F.) for temperature are not exceeded. These limits are based on the capabilities of commercial conditioning equipments and, while generally acceptable for acceptance testing, are not close enough for referee work. In the latter case, humidity should be maintained at plus or minus 1 per cent and temperature at plus or minus 0.5 C. (1 F.) or at closer limits, if obtainable, as agreed upon by the manufacturer and the purchaser. In small chambers, such as desiccator jars using acid or salt solutions, a small leak should be provided to maintain the conditioned space at approximately atmospheric pressure. In such containers a sulfuric acid-water mixture may be used to maintain the humidity at any desired point (see Fig. 6). Saturated solutions of certain salts such as sodium chloride, potassium chloride, and sodium nitrate may also be used to maintain definite humidity conditions. The temperature of the desiccator or other container shall be maintained constant, and an adequate surface exposure of the mixture or solution shall be provided. A gentle air circulation should be maintained, but not sufficient to pick up droplets of the solution.

#### Method of Measuring Resistance

12. The resistance may be measured by means of a galvanometer used as an ammeter. The galvanometer shall have a suitable shunt for increasing its range. Figure 7 shows the diagram of connec-

tions and guarding of circuits, which are suitable for use in measuring the insulation resistance of specimens of the types shown in Figs. 1, 2 and 3, or for the approximate separation of surface and volume resistance by means of the arrangements shown in Fig. 4 or 5.

#### Insulation Resistance Determination

13. (a) The insulation resistance required in Section 10 (c) shall be determined as follows: Connect the leads to adjacent terminals on the specimen. Set the universal shunt at the position of lowest galvanometer sensitivity, close the battery reversing switch to the desired polarity. Increase the shunt ratio until a readable deflection is obtained on the galvanometer. Read the deflection 1 min. after closing the reversing switch. Open the reversing switch and close key K-1.

(b) The resistances required in Section 10 (c) and (d) shall be determined by the deflection method, using the completely guarded circuit (Fig. 7) including the guard electrode on the specimen. The procedure is the same as that specified in Paragraph (a). In either case, if no appreciable deflection is obtained when the universal shunt is set at the position of maximum galvanometer sensitivity, the resistance is too high to be measured without resorting to one of the methods mentioned in Note 1 under Section 8.

#### Precautions

14. (a) *Mounting Specimens.*—In mounting the specimens for measurement, it is important that there shall be no conductive paths between the electrodes except those through the specimen. For example, the specimens shown in Figs. 1, 2 and 3 shall be held by one or both edges so that no one of the electrodes touches the supports.



(b) *Measuring High Resistances.*—In measuring high resistances by the set-up shown in Fig. 7, it is important that all parts of the apparatus between the guarded electrode and the battery terminal of the galvanometer shall be guarded, and adequately insulated from the guard. This applies also to key, K-1. The guard shall be sufficiently insulated from ground and from contact with the operator to prevent failure of the battery by leakage and danger of shock to the operator. Apparatus between the guarded electrode and the battery terminal of the galvanometer shall be sufficiently insulated from the guard so that the leakage shall not form an appreciable shunt on the galvanometer. This insulation resistance need be only 10 megohms.

(c) *Measuring Volume Resistance.*—In measuring the volume resistance by the deflection method it is important that the current flow between the guarded electrode and the guard electrode shall be negligible. The current through the specimen divides between this path and that through the galvanometer with its shunt, and only the flow through the galvanometer is measured. In order that the error resulting from this source shall be less than 1 per cent, the insulation resistance between the guarded electrode and the guard electrode shall be more than 100 times the resistance of the galvanometer with its shunt. The key, K-2, which short circuits the calibrating resistance when it is closed, makes it possible to reduce this error by reducing the ratio of the resistance of the galvanometer path to that of the insulation resistance between guarded and guard electrodes. If, when measuring a resistance of over 100 megohms, an appreciable change of deflection is seen when key K-2 is closed or opened, it is an indication that leakage is occurring between guard and guarded

electrodes. The reading with key K-2 closed is the more nearly correct and should be used in calculating the volume resistance.

(d) *Calibration.*—In calibrating, the specimen shall be short circuited by means of key K-1, and with the same voltage applied that was applied to the specimen, the galvanometer deflection produced shall be read when only the calibration resistance is in series with it, using a suitable shunt ratio.

### CALCULATIONS

#### Volume Resistance

15. If the calibrating resistance has a sufficiently high value, and a suitable universal shunt is provided, the resistance of a specimen may be easily compared with the calibrating resistance. The shunt setting may be represented by a fractional ratio,  $s$ , in negative powers of ten, or by a multiplying factor,  $f$ , in positive powers of ten. In the former case, when measuring the specimen, call the shunt ratio,  $s$ , and the galvanometer deflection,  $d$ . Let the galvanometer deflection observed during calibration (Section 14 (d)) be represented by  $D$ , and the corresponding shunt ratio by,  $S$ ; then if the calibrating resistance has a value,  $M$ , the resistance of the specimen,  $R$ , may be calculated from the following formula:

$$R = M \cdot \frac{D}{d} \cdot \frac{s}{S} - M \dots (1)$$

Or, if the shunt setting is given in multiplying factors, the formula becomes:

$$R = M \cdot \frac{D}{d} \cdot \frac{F}{f} - M \dots (2)$$

where  $F$  = the shunt setting for the calibrating resistance, and  
 $f$  = the shunt setting for the specimen.

If the calibrating resistance is so small



that the battery will give too large a deflection of the galvanometer with the smallest shunt ratio (or largest multiplying factor), it is then necessary to calibrate by means of a low-voltage battery. The same measurements shall be made as in the previous cases, but it is now necessary to know the voltage of the calibrating battery,  $E$ , and of the measuring battery,  $e$ . Then:

$$R = M \cdot \frac{D}{d} \cdot \frac{s}{S} \cdot \frac{e}{E} - M \dots (3)$$

or

$$R = M \cdot \frac{D}{d} \cdot \frac{F}{f} \cdot \frac{e}{E} - M \dots (4)$$

### Insulation Resistance

16. The insulation resistance is the resistance as measured between any two adjacent binding posts of Fig. 1, between any two adjacent metal strips of Fig. 2, or between the tapered pins of Fig. 3. This value should be expressed merely as "insulation resistance." Where it is a certainty that the leakage is confined entirely to the surface, results so obtained may be converted to surface resistivity in terms of "surface resistance per square" by using the following relation for the disks:

$$\sigma = \frac{\pi R'}{\ln \left[ \frac{d}{2r} + \sqrt{\left( \frac{d}{2r} \right)^2 - 1} \right]} \dots (5)$$

where  $R'$  = twice the measured resistance,

$d$  = the distance between the centers of the disks,

$r$  = the radius of the disks, and

$\ln$  = the natural logarithm.

If  $d$  has a value between  $3r$  and  $4r$  the following approximate formula may be used:

$$\sigma = \frac{\pi}{2} \cdot \frac{R'(d+r)}{d-r} \dots (6)$$

In the case of metal strips applied to a

thin tape or strip of insulating material the surface resistance per square may be calculated by the following formula:

$$\sigma = \frac{R'b}{L} \dots (7)$$

where  $R'$  = twice the surface resistance as measured,

$b$  = average width of the tape or strip of insulating material, and

$L$  = distance between the electrodes.

### Volume Resistivity

17. The volume resistivity,  $\rho$ , shall be calculated from the following formula:

$$\rho = \frac{RA}{t} \dots (8)$$

where  $R$  = the volume resistance as measured with the specimen shown in Fig. 4 or 5 in accordance with Section 10 (d),

$A$  = the area of the guarded electrode, and

$t$  = the average thickness of the specimen.

### Surface Resistance

18. (a) The surface resistance may be calculated from the measurements made with the specimen shown in Fig. 4 in accordance with Section 10 (c) and (d). This is by far the most reliable method. The formula for calculating the surface resistance is as follows:

$$R' = \frac{R_s R}{R - R_s} \dots (9)$$

where  $R'$  = the surface resistance,

$R_s$  = the over-all resistance, Section 10 (c), and

$R$  = the volume resistance, Section 10 (d).

(b) *Surface Resistivity*.—The surface resistivity shall be calculated from the surface resistance as follows:

$$\sigma = \frac{R'c}{L} \dots\dots\dots (10)$$

where  $\sigma$  = the surface resistivity,  
 $R'$  = the surface resistance,  
 $c$  = the average circumference of the guarded electrode and of the inner edge of the guard electrode, and  
 $L$  = the distance between the electrodes.

#### REPORT

#### Report

19. The report shall include the in-

formation indicated in the following Paragraphs (a), (d), (e), (f), (g) and (h) and may include the items in Paragraphs (b) and (c):

(a) The over-all insulation resistance in ohms,

(b) The volume resistivity in ohm-cm. units,

(c) The surface resistivity in ohms,

(d) The temperature in degrees Centigrade,

(e) The percentage relative humidity,

(f) Time of exposure to that humidity,

(g) The voltage used when other than the specified voltage, and

(h) The type of electrodes.

# Standard Methods of

## TESTING, GRADING, AND CLASSIFYING NATURAL MICA<sup>1</sup>



A.S.T.M. Designation: D 351 - 46

ADOPTED, 1938; REVISED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 351; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover tests for the grading of all natural block mica and mica splittings for general use according to size, classification of visual and electrical quality, and thickness.

### GRADING

#### Grading According to Size

2. (a) *Grade*.—Natural block mica and mica splittings shall be graded according to the area of the usable rectangle which can be cut from the specimen and the minimum dimension of one side. The mica in the usable rectangle shall meet the quality requirements specified by the purchaser.

(b) *A.S.T.M. Chart*.—The standard A.S.T.M. chart shown in Fig. 1 (Plate I) shall be used for grading natural block mica and mica splittings according to size.

(c) *Method of Grading for Size*.—The specimen to be graded shall be laid upon

the chart so that it covers point *O* and has its maximum and minimum dimensions extending along and covering the lines *OA* and *OB*, respectively. The specimen should be shifted until its usable area completely covers the largest rectangle determined by a diagonal extending from point *O* to or beyond a point on any of the curves, Nos. 6, 5½, 5, 4, 3, 2, 1, or A-1 (Special). The number of the curve at greatest distance from *O* cut by the diagonal of the rectangle designates the grade of the specimen.

NOTE.—The A.S.T.M. chart complies closely with the following so-called India grading scale, in general use for many years:

Grade	Area of Rectangle, sq. in.	Minimum Dimension of One Side, in.
OOEE Special	100 and over	<i>a</i>
OEE Special	80 to 100	<i>a</i>
EE Special	60 to 80	<i>a</i>
Extra Special	48 to 60	<i>a</i>
A-1 (Special)	36 to 48	4
1	24 to 36	3
2	15 to 24	2
3	10 to 15	2
4	6 to 10	1½
5	3 to 6	1
5½	2½ to 3 <sup>b</sup>	1
6	1 to 2½	¾

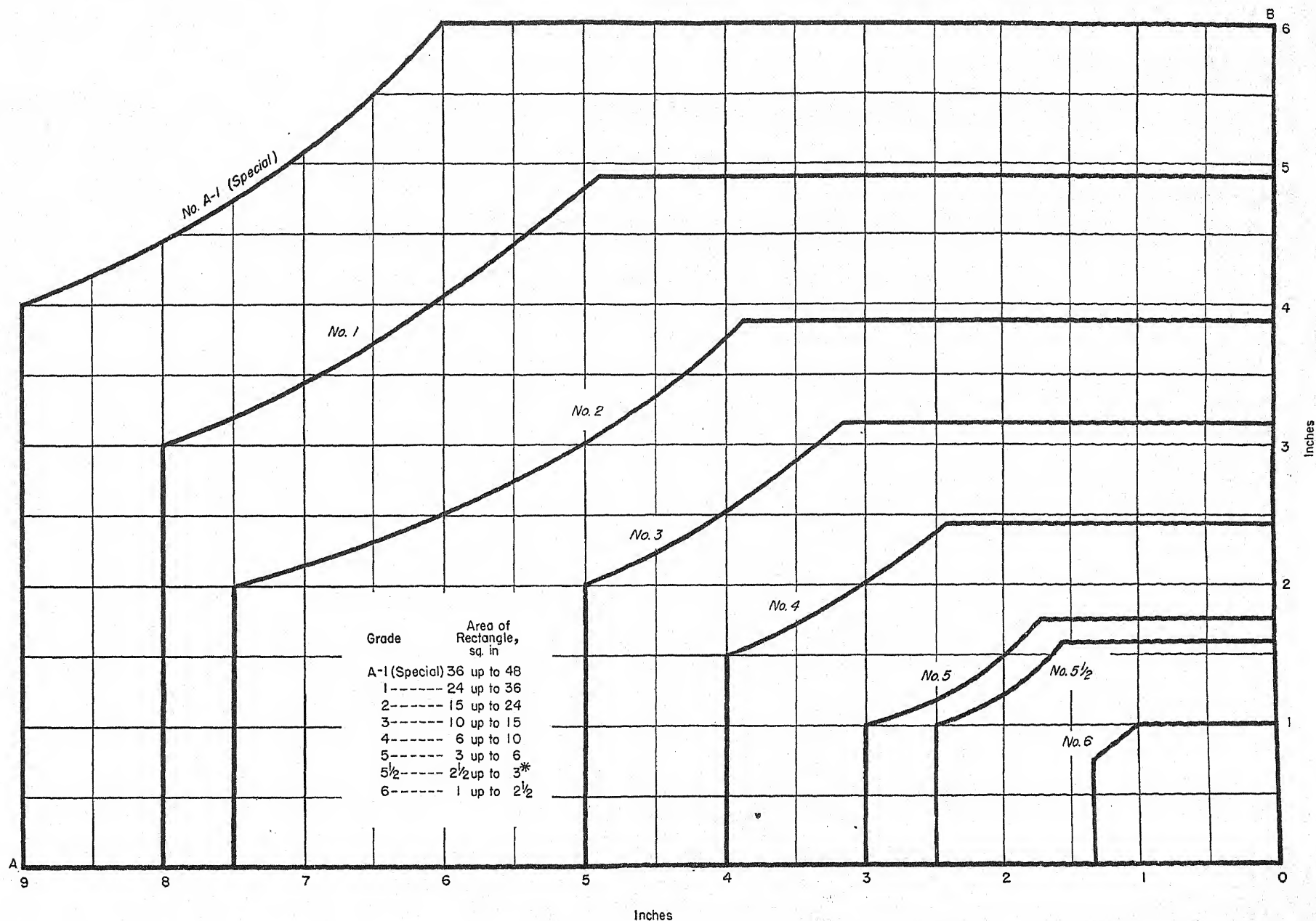
<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1932 to 1938, being revised in 1933, 1935, 1936, and 1938.

The procedure for power factor and dielectric constant was issued as tentative in 1939, revised in 1942, and adopted in 1946 with revisions.

<sup>a</sup> While grades OOEE Special, OEE Special, EE Special, and Extra Special are available commercially, there is not sufficient information available at present to establish minimum dimensions.

<sup>b</sup> 1943 India Mica Mission permits 2¼ to 3 sq. in.



\* 1943 India Mica Mission permits 2 1/2 to 3 sq. in.

FIG. 1.—A.S.T.M. Chart for Grading Natural Block Mica and Mica Splittings According to Size.

NOTE.—While grades OEEE Special, OEE Special, EE Special, and Extra Special are available commercially, there is not sufficient information available at present to establish minimum dimensions.



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## CLASSIFICATION

## Classification of Quality

3. (a) The classification of visual quality of block mica and mica splittings shall be judged in terms of the requirements specified in Tables I, II, and III.

TABLE I.—CLASSIFICATION OF QUALITY OF MICA.

Classification	Description
Clear.....	Free of all mineral and vegetable inclusions, stains, air inclusions, waves or buckles. Hard transparent sheets.
Clear and Slightly Stained.....	Free of all mineral and vegetable inclusions, cracks, waves, and buckles, but may contain slight stains and air inclusions.
Fair Stained....	Free of mineral and vegetable inclusions and cracks. Hard. Contains slight air inclusions and is slightly wavy.
Good Stained....	Free of mineral inclusions and cracks but contains air inclusions, some vegetable inclusions and may be somewhat wavy.
Stained.....	Free of mineral inclusions and cracks but may contain considerable clay and vegetable stains and may be more wavy and softer than the better qualities.
Heavy Stained.	Free of mineral inclusions but contains more clay and vegetable stains than that of Stained Quality, and distinctly inferior as regards to rigidity and toughness.
Black Stained and Spotted.....	Apt to contain some mineral inclusions consisting of magnetite (black), specularite (red), and hydrous iron oxide (yellow).

and mica splittings shall be determined by the test methods specified in Sections 5 to 10.

## Thickness

4. (a) Measurements of the thickness of mica shall be determined with a machinist's micrometer (Note 1) as specified in the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup> In determining thickness of mica which must be kept within small permissible variations or where two or more specimens are to be measured at once, Method A shall be used. Where the maximum accuracy is not required, Method B may be used.

NOTE 1.—For commercial purposes, it shall be permissible to measure the thickness of mica films or splittings with a high quality spring-loaded dial-gage micrometer. However, in cases of dispute Method A as prescribed in Paragraph (a) shall be used.

(b) Because of the abrasive nature of mica, the micrometer shall be tested frequently for conformity to the requirements specified in the Standard Methods D 374. The anvil and spindle shall be cleaned as frequently as necessary to prevent the accumulation of mica dust on their surfaces (Note 2) and to preserve the accuracy of the measurements. To clean, the micrometer shall be closed lightly on a clean sheet of bond paper and the paper moved between the surfaces.

NOTE 2.—Care must be exercised in moving from one measurement location to another to maintain the surfaces of the anvil and spindle parallel to the surfaces of the specimen at all times so as to avoid scratching the mica and accumulating mica dust under the micrometer surfaces which will cause false readings.

(b) The classification of power factor and dielectric constant of block mica

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# POWER FACTOR AND DIELECTRIC CONSTANT

## Scope

5. This method of test covers the procedure for determining the power factor

trodes under pressure or vertical mercury electrodes, conforming to the requirements of Paragraphs (b) or (c), is optional for either block mica or mica films. Greater precautions will be necessary in the use of either method in testing

TABLE II.—QUALITY CLASSIFICATION OF NATURAL BLOCK MICA 0.007 TO 0.030 IN. IN THICKNESS BASED ON VISUAL JUDGMENT.

NOTE 1.—The visual properties of block mica usually are judged under the following light conditions:

*For stains and inclusions*—Transmitted daylight or equivalent.

*For air inclusions*—Reflected daylight or equivalent.

*For waves, buckles, ridges, etc.*—Reflected daylight through a window frame where distortion of parallel and vertical lines of reflected image can be judged.

NOTE 2.—The mechanical properties of block mica usually are judged as follows:

*Hardness*—As judged by sharp clear ring when mica is dropped on a hard surface.

*Rigidity*—Relative stiffness judged by flexing with fingers.

NOTE 3.—Heavy stained and black stained and spotted qualities are obtainable in hard and soft varieties and with or without conducting veins and spots.

NOTE 4.—Natural mica occurs in various colors which are more pronounced the thicker the block. Some typical colors are ruby, white, light green, green, rum, etc.

NOTE 5.—Quality of films less than 0.007 in. in thickness shall be visual equivalent of corresponding block.

Properties.....	Clear Uniform Color	Air Inclusions				Slight Stains (Color Variation)	Light Dotted	Light Spotted	Sooty Stains	Black Stained and Spotted	Red Stained and Spotted	Green Stained and Spotted	Clay Stained	Vegetable Stained	Mineral Inclusions			Waviness		Buckles, Ridges, Reaves, etc.	Hardness			Cracks (Edge)
		None	Slight	Medium	Heavy										Slight	Medium	Heavy	Slight	Medium		Heavy	Rigid	Hard	
<i>Classifications:</i>																								
Clear.....	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Clear and Slightly Stained.....	✓	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Fair Stained.....	✓	✓	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Good Stained.....	✓	✓	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Stained (A).....	✓	✓	✓	✓	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Stained (B).....	✓	✓	✓	✓	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Heavy Stained.....	✓	✓	✓	✓	✓	✓	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×	×
Black Stained and Spotted.....	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

✓ = permissible.

X = not permissible.

\* Except in regions adjacent to edge.

TABLE III.—VISUAL QUALITY OF MICA FILMS 0.0008 TO 0.004 IN. IN THICKNESS.

Commercial Quality Designation	Visual Block Quality per A.S.T.M. Methods D 351	Requirements per Table II of A.S.T.M. Specifications D 748	
		Air Inclusions	Flatness
A.....	Fair stained (min.)	A	A
B.....	Good stained (min.)	B	A
C.....	Stained	C	B

and dielectric constant of natural block mica 0.007 to 0.030 in. in thickness and mica films or capacitor splits 0.0008 to 0.004 in. in thickness.

## Apparatus

6. (a) The use of either flat steel elec-

mica films thinner than 0.002 in. due to greater sensitivity to contamination and humidity effects.

(b) *Steel Electrodes*.—Three electrodes made of stainless steel or nickel-plated tool steel will be required. The electrodes shall be cylindrical in shape and of a diameter sufficient to provide the minimum specified capacitance (Note 1). The upper and lower electrodes shall have a minimum axial length of  $\frac{1}{2}$  in., and the center electrode shall have a maximum length of  $\frac{1}{4}$  in. A low resistance contact and conductor to the electrode is essential for power factor measurements in the order of 0.0001.

The upper and lower electrodes shall be electrically connected together, thus forming a two terminal capacitor, the center electrode serving as the active or measuring terminal. The surfaces of the electrodes, adjacent to the specimen, shall be ground and polished optically flat, also parallel to each other. The upper electrode shall be provided with a recess for a steel ball, so that the applied pressure will be uniformly distributed. The electrodes shall be carefully and accurately aligned without scratching the surface of the mica. It is recommended that a slotted V-shaped jig be used as an aid in aligning the electrodes.

NOTE 1.—Steel electrodes having diameters of  $\frac{3}{4}$ , 1,  $1\frac{1}{4}$ , and  $1\frac{1}{2}$  in. have been found satisfactory for practical thicknesses of mica specimens.

(c) *Mercury Electrodes.*—Three hollow stainless steel or nickel-plated cold-rolled steel electrodes filled with clean mercury, mounted with the axis horizontal so that the test specimens are in a vertical plane, will be required as shown in Fig. 2. The electrodes shall be cylindrical in shape and of the same outside diameter, which shall be large enough to provide the minimum specified capacitance (Note 2). Two adjustable electrodes each having an axial length of approximately  $\frac{3}{4}$  in., provided with suitable cavities, shall be mounted on screws in a solid stainless steel or nickel-plated cold-rolled steel rectangular yoke. A center or fixed electrode consisting of a hollow ring approximately  $\frac{3}{8}$  in. in length shall be mounted at the center of the steel yoke on a support of insulating material such as polystyrene, hard rubber, low-loss ceramic, or quartz. All electrodes shall taper from the inside to rather sharp edges approximately  $\frac{1}{16}$  in. in width. Two test specimens shall be clamped on either side of the center electrode, the thin electrode edges form-

ing mercury-tight seals. Then the two outer electrodes shall be filled with clean mercury introduced through a rubber tube attached to  $\frac{1}{8}$ -in. steel tubes located at the bottom of each electrode. Small vent holes shall be provided in the top of the outer electrodes to permit the escape of entrapped air as the mercury rises. The center electrode shall be filled through a  $\frac{1}{8}$ -in. steel tube projecting approximately  $\frac{1}{8}$  in. above the top of the electrode and extending three fourths of the way down inside the steel ring.

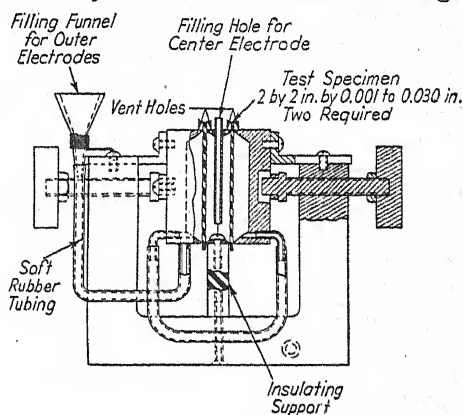


FIG. 2.—Mercury Electrode Test Assembly.

Vent holes on either side of the projecting steel tube, to permit the escape of entrapped air as the mercury rises, shall be provided. With the test specimens clamped in position, the electrodes shall be in good alignment. As in the case of the flat steel electrodes a two terminal capacitor is formed, the center electrode serving as the active or measuring terminal with the outer electrodes connected together by the steel yoke at ground.

NOTE 2.—Mercury electrodes having diameters of  $1\frac{1}{4}$  in. have been found satisfactory for mica specimens 2 by 2 in. by 0.001 to 0.030 in.

(d) *Lead-Foil Electrodes.*—The use of lead-foil electrode 0.0005 in. in thickness and 2.0 in. in diameter is satisfactory



for block mica 0.015 to 0.030 in. in thickness.

(e) *Rapid, Direct-Reading Method.*—Where classification of relative order of magnitude of power factor or its reciprocal  $Q$  value of block mica or films is desired, the rapid, direct-reading method specified in the Tentative Specifications for Natural Block Mica and Mica Films Suitable for Use in Fixed Mica-Dielectric Capacitors (A.S.T.M. Designation: D 748)<sup>3</sup> is satisfactory.

### Test Specimens

7. (a) Two similar test specimens of approximately equal and uniform thickness shall be used for each measurement when using steel electrodes or mercury electrodes (Section 6 (b) or (c)). Only one test specimen shall be tested at a time with lead-foil electrodes (Section 6 (d)). When the rapid, direct-reading method is used, test specimens shall conform to the requirements specified in A.S.T.M. Specifications D 748. The two specimens shall be obtained preferably from the same block or splitting and shall have a sufficient area and thickness to give a total capacitance of not less than 200  $\mu\text{mf}$ . A sufficient number of selected specimens shall be tested in order to give representative data.

(b) The thickness of the natural mica shall be determined in accordance with Method A of the A.S.T.M. Methods D 374.

### Preparation and Conditioning of Specimens

8. (a) The dielectric properties of mica are affected by temperature, humidity, pressure, etc. Therefore the preparation and conditioning of the specimen shall be made in the following manner:

(b) The surfaces of the specimen, with the exception of the specimens used in Section 6 (e), shall be thoroughly and carefully cleaned with a camel-hair

brush dipped in petrolic ether or vapor degreased using trichlorethelene. Subsequent to this cleaning, care shall be exercised not to contaminate the surfaces in handling.

(c) After cleaning, the specimen shall be placed in an air oven, maintained at 105 to 110 C., for a period of 1 hr. and upon removal from the oven shall be placed immediately in a desiccator and kept there until ready for test.

### Procedure

9. (a) When steel electrodes, mercury electrodes, or lead-foil electrodes (Section 6 (b), (c) or (d)) are used, the power factor and dielectric constant of the mica shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150),<sup>3</sup> except for size and type of electrode. When the rapid, direct-reading method (Section 6 (e)) is used, the power factor or  $Q$  value shall be determined in accordance with the procedure described in A.S.T.M. Specifications D 748.

(b) Certain types of micas are affected by pressure; therefore, when the flat steel electrodes specified in Section 6 (b) are used, a sufficient range of pressures (Note 1) shall be applied so that a curve of pressure in pounds per square inch *versus* power factor and dielectric constant may be obtained.

NOTE 1.—Pressures in the order of 100 to 10,000 psi. may readily be obtained by the use of an automobile type hydraulic jack equipped with a pressure gauge.

(c) Mercury electrodes specified in Section 6 (c) and lead-foil electrodes specified in Section 6 (d) give capacitance values comparable with those obtained at the highest pressures when using the flat steel electrodes (Note 2). The mercury used shall be clean and its

surface shall be bright and free of scum. Health hazard precautions should be observed when using mercury, particularly at elevated temperatures.

NOTE 2.—In order to compare satisfactorily the power factor and dielectric constant of various specimens of mica, it may be necessary to investigate such properties over a wide frequency range. However, it is recommended that at least one measurement be made at 1000 kilocycles and a temperature of  $25 \pm 5$  C. If the flat steel electrodes are used, the pressure for this measurement shall be 1000 psi.

### Calculations

10. Since two specimens are used in each measurement when using steel electrodes or mercury electrodes, the equivalent "parallel thickness" shall be used in calculating the dielectric constant. This is obtained as follows:

$$T_e = \frac{1}{\frac{1}{t_1} + \frac{1}{t_2}}$$

where:

- $T_e$  = equivalent parallel thickness,  
 $t_1$  = thickness of upper specimen,  
 and  
 $t_2$  = thickness of lower specimen.

### Report

11. The report shall include the following:

- (a) Description of mica.
- (b) Test conditions:
  - (1) Frequency in kilocycles,
  - (2) Temperature in degrees Centigrade,
  - (3) Pressure in pounds per square inch when flat steel electrodes are used,
  - (4) Voltage stress in volts per mil,
  - (5) Relative humidity in per cent, and
  - (6) Type of electrodes and diameter in inches.
- (c) Specimen data:
  - (1) Capacitance in micro-microfarads,
  - (2) Equivalent parallel thickness, and
  - (3) The power factor and dielectric constant for each value of pressure.
- (d) Method of measurement.

# Standard Methods of TESTING PASTED MICA USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 352 - 39

ADOPTED, 1936; REVISED, 1939.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 352; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods are intended to apply to the testing of mica sheets built up from mica splittings, bonded with an organic binder, to be used for hot molding, commutator insulation, heating plates, and other similar insulating purposes.

## DECREASE IN THICKNESS UNDER PRESSURE

### Apparatus

2. The apparatus shall consist of the following:

(a) *Clamp*.—A clamp for holding the test specimen of mica as illustrated in Fig. 1.

(b) *Oven*.—An oven to obtain a temperature of  $160 \pm 5$  C. ( $320 \pm 9$  F.).

(c) *Hydraulic Press*.—A hydraulic press to obtain a pressure of 2000 psi. on the mica.

(d) *Gage*.—A micrometer gage to meas-

ure the thickness of the specimen to the nearest 0.001 in.

(e) *Thermocouple*.—A thermocouple or other temperature measuring device.

### Test Specimen

3. The test specimen shall consist of sufficient pieces, 3 by 2 in. in size, to form a stack approximately 1 in. in thickness.

### Procedure

4. (a) The specimen shall be placed in the clamp, Fig. 1, which is held together by means of bolts through the end holes. The clamp with specimen while cold shall be placed in the hydraulic press and a pressure of 100 psi. applied to the surface of the specimen. The thickness of the stack at the four corners shall be carefully determined by means of a micrometer gage, the average of these readings being taken as representing the thickness of the specimen.

(b) The bolts of the clamp shall then be tightened while the specimen is under pressure so as to maintain the approximate pressure applied by the hydraulic press as described in Paragraph (a).

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1932 to 1936.

The specimen with clamp attached shall be placed in the oven at a temperature of  $160 \pm 5$  C. ( $320 \pm 9$  F.) and allowed to remain for 5 min. after reaching the oven temperature. A thermocouple placed in the specimen is suggested for accurately measuring the temperature.

(c) The specimen with clamp attached shall then be transferred as quickly as possible to the hydraulic press and a pressure of 2000 psi. on the surface of the

## STABILITY UNDER HEAT AND PRESSURE

### Purpose

6. The purpose of this test is to determine any tendency which the layers of mica may have to slip on each other, and whether the binder will be squeezed out when the sheet is subjected to heat and pressure.

### Apparatus

7. The apparatus shall consist of the following:

(a) *Sheet Steel*.—Eleven pieces of smooth sheet steel, 3 by 2 by  $\frac{1}{8}$  in.,

(b) *Clamp*.—A clamp for holding the mica as illustrated in Fig. 1.

(c) *Oven*.—An oven to obtain a temperature of  $160 \pm 5$  C. ( $320 \pm 9$  F.).<sup>2a</sup>

(d) *Hydraulic Press*.—A hydraulic press to obtain a pressure of 4400 psi. on the mica.

(e) *Thermocouple*.—A thermocouple or other temperature measuring device.

### Test Specimen

8. The test specimen shall consist of ten pieces of mica, 3 by 2 in. by the thickness of mica.

### Procedure

9. (a) Alternate layers of mica and pieces of sheet steel shall be stacked, and each stack shall have a steel sheet on the top and bottom.

(b) The specimen shall be placed in the clamp, Fig. 1, which is held together by means of bolts through the end holes. The clamp with specimen while cold shall be placed in the hydraulic press and a pressure of 100 psi. applied to the surface of the specimen. The bolts of the clamp shall then be tightened while the specimen is under pressure.

(c) The specimen with clamp attached shall be placed in the oven at a temperature of  $160 \pm 5$  C. ( $320 \pm 9$  F.).<sup>2a</sup>

<sup>2a</sup> Editorially revised in November, 1945; temperatures were formerly incorrectly specified as 230 C. (446 F.).

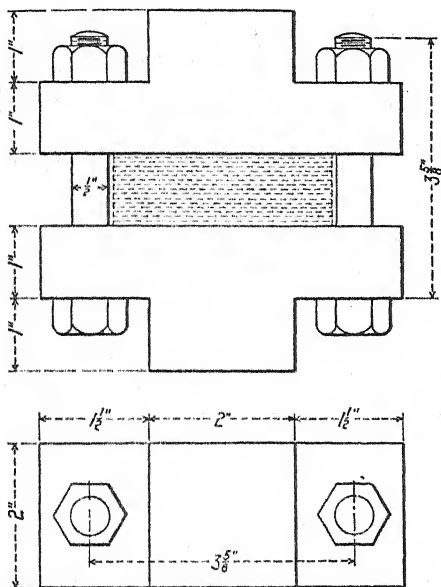


FIG. 1.—Clamp for Holding Test Specimen.

specimen shall be applied immediately and maintained until the temperature of the specimen reaches the temperature at which the original thickness was determined. The thickness of the stack shall then be determined as described in Paragraph (a).

### Report

5. The report shall include the following:

(a) The thickness of the specimen before and after applying pressure, and

(b) The percentage reduction in thickness, based on the original thickness.

and allowed to remain 5 min. after reaching the oven temperature. A thermocouple is suggested for accurately measuring the temperature of the specimen.

(d) The specimen with clamp attached shall then be transferred as quickly as possible to the hydraulic press and a minimum pressure of 4400 psi. on the surface of the specimen shall be applied immediately and maintained for 30 min.

(e) The mica in the stack shall be observed when the specimen is taken from the oven, when the hydraulic pressure is first applied, and during the period in the hydraulic press, especially during the first 5 or 10-min. period.

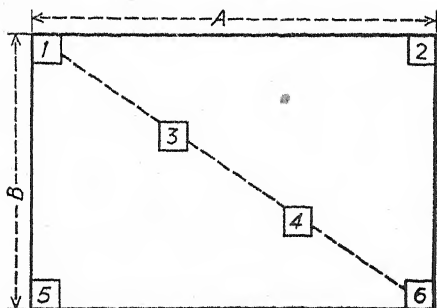


FIG. 2.—Pattern for Location of Test Pieces.<sup>3</sup>

Minimum size of sheet: A = 36 in.; B = 18 in.

### Report

10. The report shall include the following:

- (a) Whether the mica laminations slip or become displaced, and
- (b) Whether any binder exudes.

### MICA OR BINDER CONTENT

#### Purpose

11. The purpose of this test is to ascertain the relative percentages of mica and bonding materials.

#### Apparatus

12. The apparatus shall consist of the following:

- (a) *Burner*.—Bunsen burner.
- (b) *Dishes*.—Platinum or porcelain dishes.

### Test Specimen<sup>3</sup>

13. (a) *Specimens From Sheets*.—A composite specimen from a sheet shall be composed of a number of individual pieces cut from the sheet in accordance with Fig. 2 and shall be of sufficient size to give a total weight of 5 to 10 g.

(b) *Specimens From Fabricated Parts*.—A representative test specimen weighing 5 to 10 g. shall be selected from the lot.

### Procedure

14. (a) Each specimen shall be weighed to the nearest 0.0001 g. in a tared dish.

(b) The dish with the specimen shall be placed over a bunsen burner and heated at a low red heat (to avoid the dehydration of mica) until all the organic material and carbon are burned off. The contents shall then be carefully agitated by moving the dish to obtain complete combustion. After cooling in a desiccator, the weight of the residue shall be determined.

### Report

15. The report shall include the percentage loss in weight of the specimen indicated as binder and the percentage of residue in the crucible indicated as mica.

### MOLDING TEST

#### Purpose

16. The purpose of the molding test is to measure the ability of the sheet to hold its shape when molded.

#### Apparatus

17. (a) For specimens having a thickness of  $\frac{1}{16}$  in. and less, a mandrel having a diameter of  $1\frac{1}{2}$  in. shall be used.

(b) For specimens over  $\frac{1}{16}$  in. up to and including  $\frac{3}{32}$  in. in thickness a mandrel having a diameter of 2 in. shall be used.

<sup>3</sup> Editorially revised in February, 1947.

### Test Specimen

18. The test specimen shall be 2 in. in width and of sufficient length to form a butt joint on the mandrel specified in Section 17 (a) or (b).

### Procedure

19. (a) The test specimen shall be heated on a hot plate or a steam table at a specified temperature between the range of 90 and 125 C. (194 and 257 F.) and then formed around a mandrel as specified in Section 17 (a) or (b). The specimen shall then be rolled on a cold surface plate until it is cooled and the mandrel then removed.

### Report

20. The report shall include the following:

(a) Original thickness of the test specimen in thousandths of an inch,

(b) Whether the mica sheet flakes and buckles, and

(c) Whether the specimen retains its molded form.

### DIELECTRIC STRENGTH

#### Dielectric Strength

21. Except as specified below in Section 22, the dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>4</sup>

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Procedure

22. (a) Tests shall be made by either the short-time method or the step-by-step method.

(b) In tests made by the short-time method, voltage shall be increased at the rate of 0.5 kv. per sec.

(c) In tests made by the step-by-step method, each step shall be of 1-min. duration. An initial voltage in the short-time test, shall be adjusted as shown in the following table:

Breakdown Voltage by Short-Time Method	Adjust 50 per cent of Breakdown Voltage to the Nearest	Increment of Increase of Test Voltage
25 kv. or less . . . .	1.0 kv.	1.0 kv.
Over 25 to 50 kv., incl. . . . .	2.0 kv.	2.0 kv.
Over 50 to 100 kv., incl. . . . .	5.0 kv.	5.0 kv.
Over 100 kv. . . . .	10.0 kv.	10.0 kv.

The voltage shall then be increased in equal increments as stated in the above table. The change from each step to the next higher shall be made as rapidly as possible, and the time to change included in the succeeding test interval.

### RESISTIVITY

#### Resistivity

23. The resistivity shall be determined in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257) of the American Society of Testing Materials.<sup>4</sup>

# *Standard Methods of*

## MEASURING MICA STAMPINGS USED IN ELECTRONIC DEVICES AND INCANDESCENT LAMPS<sup>1</sup>



A.S.T.M. Designation: D 652 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 652; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover the procedures for measuring the hole spacing, thickness, and hole size of small pieces of fabricated natural mica such as bridges, spacers, and supports used in electronic devices and incandescent lamps.

### HOLE SPACING

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Microscope*.—A toolmaker's microscope, comparator, or any other traveling stage microscope conforming to the following requirements:

- (1) Magnification of approximately 40 $\times$ ,
- (2) Ocular equipped with crosslines, and
- (3) Stage equipped with one or more micrometers for movement in a horizontal plane. The micrometers shall be graduated to 0.0001 in. for direct reading.

NOTE.—It is preferable for fast checking to use a microscope having a stage equipped with two micrometers which travel at right angles to each other.

(b) *Illumination*.—A light source, which shall be reflected upward through the bottom of the microscope through the slide stage and into the field of vision.

#### Test Specimens

3. At least two test specimens shall be selected from each package of 5000 stampings or fraction thereof. If two or more dies have been used in fabricating stampings from the same type of mica, each group of stampings shall be kept separate and labeled so that separate measurements may be made on specimens from each group.

#### Procedure

4. (a) If the measuring device is equipped with two micrometers, the mica stamping to be measured shall be placed on the traveling slide stage and the microscope focussed on the specimen. The specimen shall be carefully lined up with the crosslines of the microscope in accordance with its horizontal axis. It will usually be found convenient to

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1941 to 1943.

select two holes on the same axis which are on the extreme side of the specimen and center these holes with the horizontal crossline of the eyepiece. By means of the cross slide movement, one side of the image of the object to be measured shall be brought into coincidence with the crossline of the eyepiece and a reading of the micrometer screw taken. The stage with the specimen shall then be moved by means of the micrometer screw until the crossline is in coincidence with the next point of measurement (Note 1) and a reading again taken of the micrometer screw. The difference between the first and second readings gives the actual linear distance between the two points. The procedure shall be repeated between all points which are to be measured that can be covered by the movement of the one micrometer screw (Note 2).

(b) If the measuring device is equipped with only one micrometer, the specimen may be measured as described in Paragraph (a) in one direction and then rotated 90 deg. and measured in a second direction (Note 1).

NOTE 1.—Spacings on mica stampings for electronic devices are usually specified from the center of one hole to the center of another. The center of a hole shall be determined as the point midway between the maximum diameter which can be measured across the hole in the direction of measurement. In determining the maximum diameter of a hole which shows ragged punching or burrs, the point of definite punching demarcation should be used as the boundary and the burr discounted.

NOTE 2.—The toolmaker's microscope or comparator has a maximum stage movement of 1.000 in. Distances greater than 1.000 in. shall be determined in two or more separate measurements.

(c) *Number of Measurements.*—If any one of the hole spacings of one of the specimens from a package of 5000 stampings or fraction thereof, selected in accordance with Section 3, does not agree within 0.0005 in. with the corresponding hole spacing of the second specimen

selected from the same package, two additional specimens shall be selected and measurements made on these new specimens. If all the corresponding hole spacings on three of the specimens agree within 0.0005 in., it may be assumed that the fourth specimen is erratic.

## THICKNESS

### Apparatus

5. The thickness of mica stampings shall be measured with a machinist's or barrel micrometer as described in method A of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup> For production checking, where maximum accuracy is not required, it is satisfactory to use a standard type dial gage.

### Procedure

6. Thickness measurements shall not be made over a hole or at any other portion of the mica stamping which may have a burr or ragged edge caused by fabrication. If the specimen is so small that it does not have an unfabricated area large enough to accommodate the use of a standard micrometer anvil and spindle 0.250  $\pm$  0.001 in. in diameter, a special micrometer with the anvil and spindle face small enough to make a measurement on an unfabricated area shall be used. The size of the anvil and spindle of the special micrometer shall be agreed upon by the manufacturer and the purchaser. If a small anvil and spindle micrometer is used, it shall conform to the requirements prescribed for the micrometer in Methods D 374, except for the anvil and spindle size.

## HOLE SIZE

### Apparatus

7. A tapered pin gage (Fig. 1) con-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



forming to the following requirements shall be used:<sup>4</sup>

(1) Depth dial gage with a 1-in. travel, graduated to read directly to 0.001 in., with a second hand showing each revolution of 0.100 in.

(2) The dial gage shall be a low-friction type mounted with a spindle

0.0001 in. The pin shall have a total measuring length of 1.000 in., thus the total range of any one pin will be 0.020 in.

(4) The lower anvil shall consist of a bushing  $0.250 \pm 0.002$  in. in outside diameter with a hole  $0.130 \pm 0.002$  in. in diameter.

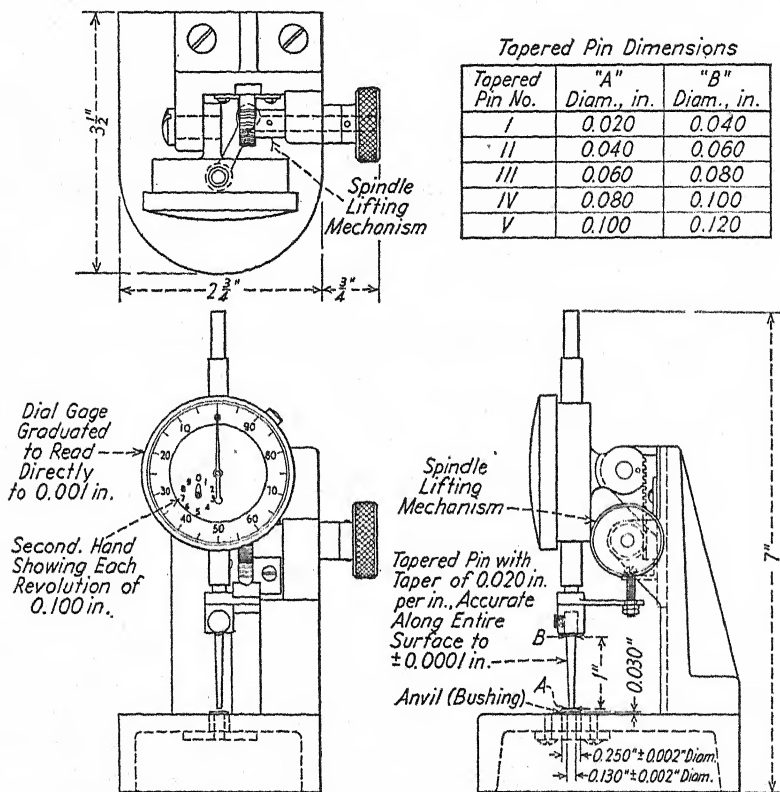


FIG. 1.—Tapered Pin Gage for Measuring the Hole Size of Mica Stampings.<sup>4</sup>

lifting mechanism of such nature that the pressure exerted on the specimen shall be not less than 1 nor more than 1.5 oz. (See Fig. 1).

(3) The tapered pin shall have a taper of 0.020 in. per in. and shall be accurate along its entire surface to plus or minus

### Test Specimens

8. Since the hole size of any given mica stamping will vary in accordance with its thickness, the specimens selected shall have a uniform thickness within plus or minus 0.001 in. of the specified mean thickness.

### Procedure

9. (a) The hole size to be measured shall be predetermined so that the ta-

<sup>4</sup> Blueprints of the detailed drawings of the tapered pin gage are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

pered pin selected will be of the proper range in diameter. The specimen shall be placed on the anvil of the gage and the pin lowered until it contacts the specimen at a point just outside of the hole to be measured. The dial reading shall then be set at zero, in order to compensate for the thickness of the mica. The specimen shall then be moved into position on the anvil so that the tapered pin will enter the hole to be measured.

TABLE I.—HOLE SIZE CONVERSION VALUES.

Dial Reading <sup>a</sup>	Hole Size, in.				
	Pin Diam-eter, 0.020 to 0.040 in.	Pin Diam-eter, 0.040 to 0.060 in.	Pin Diam-eter, 0.060 to 0.080 in.	Pin Diam-eter, 0.080 to 0.100 in.	Pin Diam-eter, 0.100 to 0.120 in.
0.050.....	0.021	0.041	0.061	0.081	0.101
0.100.....	0.022	0.042	0.062	0.082	0.102
0.150.....	0.023	0.043	0.063	0.083	0.103
0.200.....	0.024	0.044	0.064	0.084	0.104
0.250.....	0.025	0.045	0.065	0.085	0.105
0.300.....	0.026	0.046	0.066	0.086	0.106
0.350.....	0.027	0.047	0.067	0.087	0.107
0.400.....	0.028	0.048	0.068	0.088	0.108
0.450.....	0.029	0.049	0.069	0.089	0.109
0.500.....	0.030	0.050	0.070	0.090	0.110
0.550.....	0.031	0.051	0.071	0.091	0.111
0.600.....	0.032	0.052	0.072	0.092	0.112
0.650.....	0.033	0.053	0.073	0.093	0.113
0.700.....	0.034	0.054	0.074	0.094	0.114
0.750.....	0.035	0.055	0.075	0.095	0.115
0.800.....	0.036	0.056	0.076	0.096	0.116
0.850.....	0.037	0.057	0.077	0.097	0.117
0.900.....	0.038	0.058	0.078	0.098	0.118
0.950.....	0.039	0.059	0.079	0.099	0.119
1.000.....	0.040	0.060	0.080	0.100	0.120

<sup>a</sup> An 0.005 increment on dial is equivalent to 0.0001 in. on the pin diameter, and an 0.050 increment on dial is equivalent to 0.001 in. on the pin diameter.

(b) The pin shall slowly be lowered until it enters the hole and then allowed to penetrate under its own weight until the lifting mechanism indicates that it has reached its maximum distance of penetration. The dial reading shall be taken and converted to actual hole size by the conversion values shown in Table I.

(c) Slots or elongated holes which are not true circles shall be measured in the

same manner as described in Paragraphs (a) and (b), except that a correction factor of  $-0.001$  in. from the actual reading shall be used, since the pin makes contact with the mica at only two points along the minor diameter of the slot and thus tends to slightly imbed itself.

(d) Extreme care shall be taken while making measurements in order to be certain that the pressure exerted by the spindle only is used in inserting the pin into the mica stamping, otherwise the tapered pin will be forced into the hole further under additional pressure and yield very inaccurate measurements (Note).

NOTE.—The dial gage should be equipped with a counter-clockwise dial and should have the spring removed so that the spindle is suspended against the lifting mechanism only by its own weight plus that of the tapered pin. When the tapered pin engages the hole being measured, the lifting mechanism (see Fig. 1) will disengage from the spindle assembly.

(e) *Number of Measurements.*—The average of five measurements taken on five separate mica stampings shall be considered the actual hole size, the latter being calculated in accordance with the conversion values shown in Table I.

## REPORT

### Report

10. The report shall include the following:

(1) Average, minimum, and maximum hole spacing in inches,

(2) Average, minimum, and maximum thickness in inches, and

(3) Average, minimum, and maximum hole sizes in inches together with the tapered pin range size.

# STANDARD SPECIFICATIONS AND METHODS OF TEST FOR ASBESTOS YARNS<sup>1</sup>



A.S.T.M. Designation: D 299 - 42

ADOPTED 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 299; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These specifications define asbestos yarn, prescribe the methods of testing, and stipulate tolerances.

## Definitions

2. (a) *Asbestos Yarn*.—Asbestos yarn is yarn consisting of: (1) asbestos fiber, (2) asbestos and other fibers, (3) asbestos and other fibers and wire, or (4) asbestos and other fibers with an insert of cotton or other yarn reinforcement.

(b) *Plain Asbestos Yarn*.—Plain asbestos yarn is yarn consisting of: (1) asbestos fiber, or (2) asbestos and other fibers.

(c) *Metallic Asbestos Yarn*.—Metallic asbestos yarn is yarn consisting of plain asbestos yarn with an insert of brass, copper, or other fine wire. It is also termed "wire-inserted yarn."

(d) *Reinforced Asbestos Yarn*.—Reinforced asbestos yarn is plain yarn with an insert of yarn made of other fibers.

(e) *Standard Condition*.—Standard condition of asbestos yarn shall be that reached by the yarn when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

NOTE.—For purpose of tests, it is recognized that specimens reach moisture equilibrium after exposure for at least 4 hr., unless otherwise specified, under the above conditions.

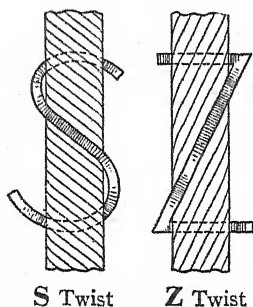
(f) *Direction of Twist*.—The direction of twist shall be as defined in the Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123) of the American Society for Testing Materials, as follows:

*Twist, Direction of*.—A yarn or cord has S twist if, when held in a vertical position, the spirals conform in direction

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to their present adoption as standard, these specifications were published as tentative from 1928 to 1929. They were adopted in 1929, published as standard from 1929 to 1941, being revised in 1933, 1936, and 1937, but withdrawn and replaced in 1941 by Tentative Specifications D 299-40 T which was published as tentative from 1940 to 1942.

of slope to the central portion of the letter "S," and Z twist if the spirals conform in direction of slope to the central portion of the letter "Z."



(g) *Number (Cut) (Asbestos).*—A term indicating the number of an asbestos yarn. The word "cut" preceded by a number indicates, in multiples of 100 yd., the yardage per pound of single-ply yarn. For example, "5-cut" indicates that a pound of single-ply yarn so designated measures approximately 500 yd.; "10-cut" indicates a yarn that measures approximately 1000 yd. per lb.

(h) *Ply.*—Ply is the number of strands of single asbestos yarn twisted together to form a heavier yarn.

*Example.*—"2-ply 10-cut" indicates a yarn of two strands, each strand being 10-cut.

### Breaking Strength

3. The average breaking strength of asbestos yarn shall be not less than that specified.

### Number (Cut)

4. (a) The standard number of asbestos yarn is designated as "cut." The yards per pound of the standard cuts shall conform to the following requirements:

Cut Designation	Nominal Yards per Pound	Permissible Variations in Average Yards per Pound	Permissible Variations in Yards per Pound in Single Skein
5-cut.....	500	450 to 549	400 to 599
6-cut.....	600	550 to 649	500 to 699
7-cut.....	700	650 to 749	600 to 799
8-cut.....	800	750 to 849	700 to 899
9-cut.....	900	850 to 949	800 to 999
10-cut.....	1000	950 to 1049	900 to 1099
12-cut.....	1200	1150 to 1249	1100 to 1299
14-cut.....	1400	1300 to 1499	1200 to 1599
16-cut.....	1600	1500 to 1699	1400 to 1799
18-cut.....	1800	1700 to 1899	1600 to 1999
20-cut.....	2000	1900 to 2099	1800 to 2199
22-cut.....	2200	2100 to 2299	2000 to 2399
24-cut.....	2400	2300 to 2499	2200 to 2599
26-cut.....	2600	2500 to 2699	2400 to 2799
28-cut.....	2800	2700 to 2899	2600 to 2999
30-cut.....	3000	2900 to 3099	2800 to 3199
35-cut.....	3500	3300 to 3699	3100 to 3899

(b) The cut of reinforced asbestos yarn shall be determined without removing the reinforcing core yarn.

(c) The nominal yardage per pound of plied asbestos yarn shall be determined in the following manner: Divide the nominal single yarn yardage per pound by the number of plies and then reduce the yardage so obtained by 10 per cent to correct for contraction caused by twisting.

(d) The yards per pound of metallic asbestos yarn is less than that of plain yarn of the same cut in proportion to the weight of the inserted wire.

(e) The yards per pound of sized yarn, single or plied, shall be subject to an additional tolerance of minus 2 per cent in comparison with the corresponding untreated yarn.

### Size of Wire

5. The diameter of the wire used in metallic asbestos yarn shall not vary from the size specified by more than plus or minus 0.001 in.

### Twist

6. The directions and turns per inch of twist shall be mutually agreed upon by the manufacturer and the purchaser.

### Grades

7. (a) The grades of plain, metallic, and reinforced asbestos yarn shall be determined by the percentage of asbestos content by weight and shall conform to the following requirements:

Grade	Asbestos Content
Commercial Grade.....	75 to 80 per cent, excl.
Underwriters' Grade.....	80 to 85 per cent, excl.
Grade A.....	85 to 90 per cent, excl.
Grade AA.....	90 to 95 per cent, excl.
Grade AAA.....	95 to 99 per cent, excl.
Grade AAAA.....	99 to 100 per cent, incl.

(b) The asbestos content of reinforced asbestos yarn shall be determined without removing the reinforcing core yarn.

(c) The asbestos content of metallic asbestos yarn shall be determined after the wire has been removed.

### METHODS OF TESTING

#### Test Conditions

8. All test specimens shall be brought to a standard condition in accordance with Section 2 (e) prior to testing and all tests shall be made under the standard condition.

#### Breaking Strength

9. (a) *Testing Machine.*—The breaking strength shall be determined on a tension testing machine conforming to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76) of the American Society for Testing Materials.<sup>3</sup>

(b) *Procedure.*—All tests shall be made on single strands. Single strands shall be broken after conditioning the tubes or bobbins for 12 hr. in accordance with Section 8. A single-strand tester of proper capacity with the clamps set 10 in. apart and having a speed of pulling clamp of  $12 \pm \frac{1}{2}$  in. per min. shall be used. The average of four breaks from each of ten tubes or bobbins selected at random from each case of

300 lb. or less shall be the average strength.

#### Number (Cut)<sup>4</sup>

10. The cut of plain or reinforced asbestos yarn, single or plied, shall be calculated by dividing by 100 the yards per pound as determined in accordance with Paragraphs (a) to (c).

(a) In determining the yards per pound of single-ply, plain or reinforced asbestos yarn there shall be weighed from each case of 300 lb. or less three 15-yd. skeins from each of 5 tubes selected at random. The skeins shall be conditioned for at least 4 hr. in accordance with Section 8 and the weight shall then be determined to the nearest 0.1 g. The yardage shall be calculated as follows:

$$\text{Yards per pound} = \frac{453.6}{\text{wt. of 15 skeins}} \times 225$$

(b) To determine the yards per pound of a single-ply yarn in plied, plain or reinforced asbestos yarn the skeins shall be selected and tested in accordance with Paragraph (a). The yardage shall then be calculated as follows:

$$\text{Yards per pound, single ply} = \frac{453.6}{\text{wt. of 15 skeins}} \times \frac{225 \times \text{no. of plies}}{0.90}$$

NOTE.—The factor 0.90 in the formula compensates for an assumed contraction of 10 per cent incurred in twisting.

(c) To determine the yards per pound of metallic asbestos yarn, single or plied, the weight in grams of the wire or reinforcing strands in a 15-yd. skein shall be determined. Twelve times this weight shall be subtracted from the weight of the twelve 15-yd. test skeins. The yardage shall then be determined for single-ply yarn in accordance with Paragraph (a) and for plied yarn in accordance with Paragraph (b).

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

<sup>4</sup> Editorially revised in April, 1944.

### Twist

11. (a) *Direction of Twist*.—The direction of twist shall be determined in accordance with Section 2 (f).

(b) *Turns in Single Yarn*.—The turns per inch of twist in single yarn shall be determined on any standard single yarn twist tester. The first 2 yd. of any package of yarn shall be discarded and all possible care shall be exercised in handling samples to prevent a change in the existing twist. The yarn, drawn just taut enough to prevent sagging, shall be gripped in the twist counter with clamps set 4 in. apart. One clamp shall be rotated until the twist is removed as indicated by the yarn breaking under its own weight or by visual examination of the sample. The twist of yarn in a shipment shall be the average of four tests from each of the ten tubes or bobbins used for determining breaking strength.

(c) *Turns in Plied Yarn*.—The turns per inch of twist in plied yarn shall be determined as described in Paragraph (b) with the following exceptions: The clamps shall be set 10 in. apart and a tension equal to the weight of 100 yd. of yarn shall be applied.

### Asbestos Content

12. One test specimen weighing not less than 5 g. shall be taken from each of the tubes used for determining number and dried to constant weight in an oven at 105 to 110 C. (220 to 230 F.), and the weights of the dried specimens recorded. The specimens shall be placed in an electric oven and heated for not less than 1 hr. at 800 to 810 C. (1470 to 1490 F.). After removal from the oven they shall be cooled in a desiccator to room temperature, and then weighed. The weight of the residue shall be divided by the factor 0.86<sup>a</sup> to determine the original weight of the asbestos content. This weight of asbestos content shall be divided by the weight of the dried specimens to obtain the percentage of asbestos. The average of the five determinations shall be the asbestos content.

NOTE.—Specimens that are soiled, or have been sized, or otherwise treated, shall be prepared for testing by removing hydrocarbon materials with chloroform, and then boiled for 5 min. in distilled water. The specimens shall then be tested in accordance with the above procedure.

<sup>a</sup> This factor is based upon a proved average of 14 per cent water of crystallization in chrysotile asbestos.

STANDARD SPECIFICATIONS AND METHODS OF TEST  
FOR  
ASBESTOS ROVING FOR ELECTRICAL PURPOSES<sup>1</sup>



A.S.T.M. Designation: D 375 - 44

ADOPTED, 1936; REVISED, 1937, 1941, 1942, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 375; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

Scope

1. (a) These specifications and methods of test apply to asbestos roving, an asbestos textile product to be used as insulation on heat-resisting fixture wire, flexible cord or heater cord, and similar electrical conductors. Two types are covered as defined in Section 2 (a) and (b).

(b) Roving, in the asbestos textile industry, is an assemblage of carded fibers rubbed into a single strand without twist.

Definitions

2. (a) *Asbestos Roving*.—Asbestos roving is composed of a mixture of chrysotile asbestos and cotton or other organic fiber.

(b) *Reinforced Asbestos Roving*.—Reinforced asbestos roving is composed of a core of yarn consisting of organic fiber, covered with a mixture of chrysotile asbestos and organic fiber.

(c) *Standard Condition*.—Standard condition of asbestos roving shall be that reached by the roving when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

NOTE.—For purpose of tests, it is recognized that specimens reach moisture equilibrium after exposure for at least 4 hr., unless otherwise specified, under the above conditions.

(d) *Number (Cut) (Asbestos)*.—A term indicating the number of an asbestos roving. The word "cut" preceded by a number indicates, in multiples of 100 yd., the yardage per pound of roving. For example, "5-cut" indicates that a pound of roving so designated measures approximately 500 yd.; "10-cut" indicates a roving that measures approximately 1000 yd. per lb.

Number (Cut)

3. (a) The standard number of asbestos roving is designated as "cut." The yards per pound of the standard

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1933 to 1936, being revised in 1934 and 1936.

cuts shall conform to the following requirements:

Cut Designation	Nominal Yardage per Pound	Permissible Variations in Yards per Pound, Average of 10 Tests	Permissible Variations in Yards per Pound, Single Tests
5.....	500	450 to 549	400 to 599
6.....	600	550 to 649	500 to 699
7.....	700	650 to 749	600 to 799
8.....	800	750 to 849	700 to 899
9.....	900	850 to 949	800 to 999
10.....	1000	950 to 1049	900 to 1099
12.....	1200	1150 to 1249	1100 to 1299
14.....	1400	1300 to 1499	1200 to 1599
16.....	1600	1500 to 1699	1400 to 1799
18.....	1800	1700 to 1899	1600 to 1999

NOTE.—The tolerances on special numbers such as 5½-cut, 6½-cut, etc. shall be equal to those of the standard numbers.

(b) The cut of reinforced asbestos roving shall be determined without removing the reinforcing core yarn.

### Grades

4. (a) The grade of asbestos roving shall be determined by the percentage of asbestos by weight, and shall conform to the following requirements:

Grade	Asbestos Content
Commercial Grade....	75 to 80 per cent, excl.
Underwriters' Grade..	80 to 85 per cent, excl.
Grade A.....	85 to 90 per cent, excl.

NOTE.—The 16 and 18 cut rovings are available only in Commercial and Underwriters' grades; all other cuts are available in all three grades.

(b) The asbestos content of reinforced asbestos roving shall be determined without removing the reinforcing core yarn.

### Iron Content

5. Roving shall be made in two varieties, ferrous and non-ferrous, the magnetic ratings of which shall conform to the following requirements:

	Total Iron Content, max., per cent	Magnetic Rating, max., per cent
Ferrous roving.....	6	2
Non-ferrous roving:		
Underwriters' Grade..	1.75	0.75
Grade A.....	2	1

## METHODS OF TESTING

### Sampling

6. (a) Each shipment shall be divided into lots of from 800 to 1000 lb. From each lot or fraction thereof, six cones, tubes, or cheeses of roving shall be taken at random and the tests prescribed shall be made on specimens removed from each package selected.

(b) If the tests from any lot fail to fall within the specified limits, a second set of samples shall be taken from another part of the lot and tested as before. The results of the two groups of tests shall be averaged to obtain the result for the lot.

(c) If the average values determined in accordance with Paragraph (b) fail to fall within the specified limits, the previous test results shall be discarded and a third set of samples shall be taken by selecting at random six cones, tubes, or cheeses from each 150 to 200-lb. unit of the material. The average results of tests on these six packages shall be considered the average of the particular unit tested, and each unit shall be accepted or rejected on this basis.

### Test Conditions

7. All test specimens shall be brought to a standard condition in accordance with Section 2 (c) prior to testing and all tests shall be made under the standard condition.

### Number (Cut)

8. (a) *Number of Tests.*—Ten determinations of the number of yards per pound shall be made on each set of six packages selected. Individual test results and the average value of the ten tests shall fall within the respective permissible variations prescribed in the table given in Section 3 (a).

(b) *Procedure.*—The packages shall be grouped on the floor and six strands shall be drawn from them slowly, looping



over a support to assemble 24 strands, which shall be cut into a 1-yd. length while suspended. The 24 yd. shall be weighed to the nearest 0.1 g. and the number of yards per pound calculated from the following formula:

$$Y = \frac{453.6 \times 24}{W}$$

where:

$Y$  = yards per pound, and  
 $W$  = weight of 24 yd. in grams.

### Asbestos Content

9. Two specimens, weighing not less than 5 g. each shall be taken from each lot or unit sampled and dried to constant weight in an oven at 105 to 110 C. (220 to 230 F.), and the weights of the dried specimens recorded. The specimens shall be placed in an electric oven and heated for not less than 1 hr. at 800 to 810 C. (1470 to 1490 F.). After removal from the oven they shall be cooled in a desiccator to room temperature, and then weighed. The weight of the residue shall be divided by the factor 0.86<sup>a</sup> to determine the original weight of the asbestos content. This weight of asbestos content shall be divided by the weight of the dried specimens to obtain the percentage of asbestos. The average of the two determinations made on the specimens from a given lot or unit shall be the asbestos content of that lot or unit.

NOTE.—Specimens that are soiled, or have been sized, or otherwise treated shall be prepared for testing by removing hydrocarbon materials with chloroform, and then boiled for 5 min. in distilled water. The specimens shall then be tested in accordance with the above procedure.

### TOTAL IRON CONTENT

#### Test Specimens

10. One specimen, weighing not less than 1 g. shall be taken from each

sample and the average of the total iron content determinations, shall be taken as the accepted determination.

### Method A

#### Reagents

11. (a) *Stannous Chloride Solution*.—Dissolve 15 g. of c.p. tin in 350 ml. of hot HCl (sp. gr. 1.19) and dilute to 1 liter.

NOTE.—A more convenient alternative method of preparing this solution is as follows: Dissolve 14.5 g. of stannous chloride in 165 ml. of HCl (sp. gr. 1.19) and dilute to 500 ml. This solution should be kept in a tightly stoppered bottle in contact with a stick of metallic tin in order to prevent oxidation. One milliliter of this solution will reduce about 0.015 g. of ferric iron to the ferrous state. The concentration of this solution will naturally increase as the tin dissolves in the acid liquor.

(b) *Mercuric Chloride Solution*.—Prepare a saturated solution containing 60 to 100 g. of  $\text{HgCl}_2$  per liter. Approximately 1.2 ml. of this solution will oxidize the tin in 1.0 ml. of the original strength stannous chloride solution.

(c) *Diphenylamine Indicator*.—Dissolve 1 g. of diphenylamine in 100 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84).

(d) *Potassium Dichromate (0.1 N)*.—Prepare a solution of potassium dichromate of approximately 0.1 N strength as follows: Dissolve 4.9 g. of pure  $\text{K}_2\text{Cr}_2\text{O}_7$  in distilled water and dilute to 1 liter. Allow to stand several hours and then standardize against a solution containing a known amount of iron in accordance with the procedure given in Section 12 (b).

(e) *Hydrochloric Acid (1:1)*.—Mix equal volumes of HCl (sp. gr. 1.19) and distilled water. Each sample will require 50 ml. of this solution.

#### Procedure

12. (a) *Hygroscopic Moisture*.—Dry the accurately weighed specimen to constant weight in an oven at 105 to

<sup>a</sup> This factor is based upon a proved average of 14 per cent water of crystallization in chrysotile asbestos.

110 C. Transfer the specimen to a desiccator, allow to cool to room temperature and reweigh. The loss in weight is hygroscopic moisture and shall be reported as a percentage of the original weight of the specimen.

(b) *Total Iron*.—Heat the specimen gently with a bunsen burner to smoke off organic matter and then ignite in a muffle at 650 C. for 20 to 30 min. Transfer the specimen to a 200-ml. porcelain casserole and digest with 50 ml. of HCl (1:1). Without filtering, reduce the ferric iron in this hot solution by adding the stannous chloride solution drop by drop from a burette until the yellow color of the  $\text{FeCl}_3$  just disappears. The white surface of the porcelain casserole affords a convenient background for determining when sufficient stannous chloride has been added. Cool the solution to 15 C. and while stirring, add 10 ml. of  $\text{HgCl}_2$  solution. The formation of a light silky precipitate indicates that the conditions are correct. If a dark precipitate of metallic mercury is formed, it indicates that an excess of stannous chloride has been added and the determination shall be repeated using the correct amount of the reducing reagent. Stir the solution vigorously for 1 min., add 3 drops of indicator solution, and dilute to 150 ml. with cold distilled water. Titrate with standard 0.1 *N*  $\text{K}_2\text{Cr}_2\text{O}_7$  having a titer no greater than 0.004 g. of  $\text{Fe}_2\text{O}_3$ . The end point is reached when a drop of the potassium dichromate solution produces an intense deep-blue coloration that remains unchanged on the further addition of dichromate.

#### Calculation and Report

13. Report the total iron as a percentage of the weight of the original specimen as calculated from the following formula:

$$\text{Total iron, per cent} = \frac{M \times N \times 0.05584}{W} \times 100$$

where:

*M* = milliliters of the standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution,

*N* = normality of the standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution, and

*W* = weight of the original specimen.

#### Method B

##### Reagents

14. (a) *Phenanthroline Indicator*.—The solution may be purchased ready for use.

(b) *Hydrochloric Acid (1:10)*.—Mix 1 volume of HCl (sp. gr. 1.19) and 10 volumes of distilled water. Each specimen will require 100 ml. of this solution.

(c) *Ceric Ammonium Sulfate Solution (0.1 N)*.—Dissolve 80 g. of ceric ammonium sulfate ( $\text{Ce}(\text{SO}_4)_2 \cdot 2(\text{NH}_4)_2\text{SO}_4 \cdot 2\text{H}_2\text{O}$ ) in 500 ml. of a  $\text{H}_2\text{SO}_4$  solution (prepared by diluting 28 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) with distilled water), stir until dissolved, and then dilute to 1000 ml. Standardize the solution, which is approximately 0.1 *N*, against a solution containing a known amount of iron in accordance with the procedure described in Section 15 (b).

##### Procedure

15. (a) *Ash*.—Dry a sample of approximately 1 g. to constant weight in an oven at 105 to 110 C., transfer the specimen to a desiccator, allow to cool to room temperature, and then reweigh. This is the oven-dry weight of the specimen, weight *D*. Place the specimen in a tared No. 0 porcelain crucible and ignite in a muffle furnace at approximately 900 C. for 20 to 30 min. Transfer the crucible and specimen to a desiccator, cool to room temperature, and then reweigh. The difference between this weight and the tare weight of the crucible is the weight of ash, weight *A*. The amount of ash shall be calculated as a percentage of the oven-dry weight of the specimen, as follows:

$$\text{Ash, per cent} = \frac{A}{D} \times 100$$

(b) *Total Iron*.—Transfer the ash (Paragraph (a)) to a 300-ml. glass beaker, add 25 ml. of c.p. HCl (sp. gr. 1.19), and heat to boiling. While the solution is boiling, add just a sufficient number of drops of c.p. HF to completely dissolve the iron, and then concentrate by boiling to a volume of 15 ml., cool, and dilute with 100 ml. of cold distilled water. Pass the solution through a Walden reductor, wash the reductor with 100 ml. of diluted HCl (1:10), add 15 to 20 ml. of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) and 3 drops of phenanthroline indicator to the filtrate, and then titrate with the standard ceric ammonium sulfate.

#### Calculations and Report

16. Report the total iron as a percentage of the oven-dry weight of the

specimen as calculated from the following formula:

$$\text{Total iron, per cent} = \frac{M \times N \times 0.05584}{D} \times 100$$

where:

*M* = milliliters of the standard ceric ammonium sulfate solution,

*N* = normality of the ceric ammonium sulfate solution, and

*D* = oven-dry weight of the specimen.

#### MAGNETIC IRON CONTENT

##### Procedure

17. The magnetic rating shall be determined by the Mapes method<sup>3</sup> on one specimen taken from each sample and the average shall be taken as the accepted determination.

<sup>3</sup> *Asbestos*, p. 19, October, 1932.

# STANDARD SPECIFICATIONS AND METHODS OF TEST FOR ASBESTOS TAPE FOR ELECTRICAL PURPOSES<sup>1</sup>



A.S.T.M. Designation: D 315 - 44

ADOPTED, 1933; REVISED, 1936, 1937, 1941, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 315; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These specifications and methods of test apply to asbestos tape woven from plain asbestos yarn and suitable for electrical purposes.

## TOLERANCES

### Tolerances

2. Tolerances are the limits within which the specified characteristics of the tape must come to constitute a good delivery on contract.

### Width

3. The width of the tape shall not vary from that specified by more than the following:

Specified Width, in.	Permissible Variations in Width, in
1 and under.....	$\pm \frac{1}{32}$
Over 1.....	$\pm \frac{1}{16}$

### Thickness

4. The average thickness of the tape shall not vary from that specified by more than plus or minus 0.003 in.

## Weight

5. The average weight of the tape shall be calculated in yards per pound and shall be not less than the following:

Thickness, in.	Width, in.	Weight, min., yd. per lb.
0.015.....	$\frac{1}{2}$ .....	85
	$\frac{3}{4}$ .....	65
	1.....	50
	$1\frac{1}{4}$ .....	40
	$1\frac{1}{2}$ .....	30
0.020.....	$\frac{1}{2}$ .....	70
	$\frac{3}{4}$ .....	53
	1.....	40
	$1\frac{1}{4}$ .....	32
	$1\frac{1}{2}$ .....	25
0.025.....	$\frac{1}{2}$ .....	45
	$\frac{3}{4}$ .....	35
	1.....	25
	$1\frac{1}{4}$ .....	18
	$1\frac{1}{2}$ .....	13
0.030.....	1.....	17
	$1\frac{1}{4}$ .....	$13\frac{1}{2}$
	$1\frac{1}{2}$ .....	11

## Weave

6. The tape shall be uniformly woven and shall have selvage edges.

## Construction

7. The number of ends and picks per inch shall conform to the following re-

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1929 to 1933, being revised in 1931.

quirements within plus 6 or minus 2 ends, and plus 5 or minus 1 pick:

Thickness, in.	Ends per Inch	Picks per Inch
0.015.....	30	18
0.020.....	22	18
0.025.....	27	12
0.030.....	27	12

### Breaking Strength

8. The breaking strength per inch of width of each specimen of the tape tested shall be not less than the following requirements:

Thickness, in.	Breaking Strength, min., lb. per in. of width	
	Before Heating	After Heating
0.015.....	30	15
0.020.....	35	15
0.025.....	50	25
0.030.....	85	43

### Electrical Properties

9. The number of conducting particles in a 10-yd. length of tape of 0.015 in. in thickness shall not exceed 100 per inch of width. This requirement applies to 0.015-in. type only.

### Asbestos Content

10. Asbestos tape for electrical purposes shall be Underwriters' grade, having an asbestos content by weight of 80 per cent up to but not including 85 per cent.

### Sizing

11. No sizing shall be used.

### Workmanship

12. The workmanship shall be first class as shown by even spacing in the weave, freedom from nap and from broken threads, uneven threads, and knots.

### Finish

13. The tape shall be calendered.

### Rolls

14. (a) The tape shall be wound in

continuous rolls on bushings having a length  $\frac{1}{16}$  in. shorter than the width of the tape and an inside diameter of  $\frac{3}{8}$  in. or  $\frac{1}{2}$  in. as specified by the purchaser. The bushings shall have sufficient strength to resist crushing in packing and shipment.

(b) The rolls shall each contain 36 yd. of tape.

(c) The end of the tape shall be secured with a gummed label.

## METHODS OF TESTING

### Sampling Rolls for Test

15. From each shipment of tape at least three rolls shall be selected and the tests prescribed shall be made on sample lengths cut from each roll. Failure of the first samples selected to conform to the requirements of the specifications will necessitate the selection of the same number of rolls for a second test.

### Test Condition

16. All test specimens shall be conditioned to moisture equilibrium in a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

NOTE.—For purpose of tests, it is recognized that specimens reach moisture equilibrium after exposure for at least 4 hr. at the above specified relative humidity and temperature.

### Width

17. (a) The width shall be determined by the use of a standard steel scale so graduated as to obtain the degree of accuracy specified in Section 3. The determination shall be made while the tape lies flat and without tension on a smooth surface.

(b) The width shall be taken as the average of five measurements made at least 1 yd. apart in any one roll, and the

average of the measurements shall be accepted as the width of the lot under test.

### Thickness

18. (a) The thickness shall be measured in accordance with Section 5 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39).<sup>3</sup>

(b) The thickness shall be taken as the average of ten measurements at ten different places on the sample not less than 12 in. apart, none of which shall be on a selvage edge.

### Weight

19. The weight of the tape shall be determined by accurately measuring a sample not less than 12 yd. in length and weighing it on a balance sensitive to 0.5 per cent of the weight of the specimen. The length determination shall be made with the material laid flat on a smooth surface and held taut without stretching.

### Construction

20. (a) *Ends*.—The total number of ends shall be determined regardless of the width of the tape.

(b) *Picks*.—The number of picks shall be determined in a length of 1 in.

(c) *Number of Tests*.—The number of ends shall be taken as that secured by making one determination per sample. The number of picks per inch shall be taken as the average of five counts made at five different places on the sample. An accepted thread counting magnifying glass shall be used in making the counts.

### Breaking Strength

21. (a) The breaking strength of the tape shall be determined on a testing

machine conforming to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76) of the American Society for Testing Materials.<sup>3</sup>

(b) Tests shall be made in the direction of the warp only. The specimens shall be tested in full section. The initial length of the test specimens between the clamps shall be 3 in. and the speed of the pulling clamp shall be  $12 \pm \frac{1}{2}$  in. per min.

(c) *Breaking Strength After Heating*.—The breaking strength after heating shall be determined on specimens which have been heated in an oven for 5 min. at 300 C. (570 F.).

(d) *Number of Tests*.—The breaking strength before and after heating shall each be determined on five specimens taken from each sample roll. No single test shall fall below the requirements prescribed in Section 8.

### Electrical Properties

22. The test for conducting particles shall be made by passing the tape at a rate of 2 yd. per minute under a pressure of 7 lb. per inch of width at prevailing atmospheric conditions between smooth, clean copper rollers, 2 in. in diameter, supported by free moving bearings. The rollers shall be connected in series with a 110-v., 25-w. incandescent lamp, to a 110-v., 60-cycle circuit. The number of flashes or glows of the lamp shall indicate the number of conducting particles. The tests shall be made on a 10-yd. length in each of the sample rolls, and the average of the determinations shall be taken as the number of conducting particles.

### Asbestos Content

23. One test specimen, weighing not less than 5 g. shall be taken from each sample roll, and dried to constant weight in an oven at 105 to 110 C. (220 to

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

230 F.), and the weights of the dried specimens recorded. The specimens shall be placed in an electric oven and heated for not less than 1 hr. at 800 to 810 C. (1470 to 1490 F.). After removal from the oven they shall be cooled in a desiccator to room temperature, and then weighed. The weight of the residue shall be divided by the factor 0.86<sup>a</sup> to determine the original weight of the

asbestos content. This weight of asbestos content shall be divided by the weight of the dried specimens to obtain the percentage of asbestos. The average of the determinations shall be the asbestos content.

NOTE.—Specimens that are soiled, or have been sized, or otherwise treated, shall be prepared for testing by removing hydrocarbon materials with chloroform, and then boiled for 5 min. in distilled water. The specimens shall then be tested in accordance with the above procedure.

<sup>a</sup> This factor is based upon a proved average of 14 per cent water of crystallization in chrysotile asbestos.

# Standard Specifications for

## 0.007-IN. COTTON TAPE FOR ELECTRICAL PURPOSES<sup>1</sup>



A.S.T.M. Designation: D 335 - 36

ADOPTED, 1934; REVISED, 1936.<sup>2</sup>

Reapproved in 1946 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 335; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These specifications cover 0.007-in. cotton tapes for use in the electrical industry.

### Cotton

2. (a) Tapes shall be woven uniformly from a good grade of cotton. They shall, as far as practicable, be free from "motes" or other impurities found in improperly cleaned cotton.

(b) Tapes shall be woven with a selvage edge which shall be held as close as is commercially possible to the nominal thickness of the tape.

### Sizing

3. (a) In order to process properly and give a smooth finish, manufacturers may use a small amount of sizing in the warp threads, but such sizing shall not exceed 8 per cent of the total weight.

(b) Finished tapes shall be practically neutral in reaction. Acids or alkalies shall not be present in quantities

which will cause the tape to deteriorate or cause other harm when impregnated by the purchaser.

It is recommended that the pH value lie between 6.5 and 7.5 based on a neutrality point of 7.0.

### Physical Properties

4. The tape shall conform to the following requirements subject to the tolerances specified in Sections 3 to 6 of the Standard Methods of Testing and Tolerances for Woven Tapes (A.S.T.M. Designation: D 259) of the American Society for Testing Materials:<sup>3</sup>

Width, in.	Ends, Total	Picks per Inch	Weight, min., yd. per lb.	Breaking Strength, min., lb. <sup>a</sup>
$\frac{1}{2}$ .....	30	30	350	20
$\frac{3}{4}$ .....	36	36	250	30
$\frac{7}{8}$ .....	45	30	225	35
$\frac{1}{4}$ .....	56	36	175	40
1.....	60	30	160	40
1.....	72	36	130	50
$1\frac{1}{2}$ ....	90	30	120	60
$1\frac{3}{4}$ ....	108	36	75	75

<sup>a</sup> Values are average of five tests.

### Methods of Testing

5. All test procedures shall be conducted in accordance with Sections 7

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1931 to 1934.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



to 17 of the Standard Methods of Testing and Tolerances for Woven Tapes (A.S.T.M. Designation: D 259) of the American Society for Testing Materials.<sup>3</sup>

### Winding

6. (a) Tapes shall be wound tightly on a suitable bushing and shall be well blocked to give smooth sides to the roll.

(b) Tapes shall be so rolled that the edges are not folded over.

(c) *Bushing*.—The bushing shall be flush with the edge of the tape and shall not vary from the nominal tape width by more than  $\frac{1}{32}$  in. Bushings shall not be split and shall have sufficient strength to withstand damage in handling and transit. The inside diameter of bushings shall be  $\frac{3}{8}$  in.

### Size of Rolls

7. Tapes shall be put up in rolls containing approximately 36 yd. or 72 yd., as specified.

### Splices

8. (a) The number of splices in any package of ten 36-yd. rolls shall not exceed four. For larger packages or rolls this limit shall be prorated.

(b) All splices shall be sewed and shall be smooth and flat. Splices shall not exceed in thickness twice the thickness of the tape.

### Securing Ends of Roll

9. Pastes or starch paste may be used in securing the ends of rolls. Pins shall in no case be used.

### Packing

10. Tapes may be packed 10 or 20 rolls to a package.

# *Standard Methods of*

## TESTING ASBESTOS TUBULAR SLEEVING<sup>1</sup>



A.S.T.M. Designation: D 628 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 628; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods of testing apply to woven and braided asbestos tubular sleeving.

### Sampling

2. Asbestos tubular sleeving shall be sampled in accordance with Section 10 of the Standard Methods of Testing and Tolerances for Tubular Sleeving and Braids (A.S.T.M. Designation: D 354) of the American Society for Testing Materials.<sup>2a</sup>

### Test Condition

3. All test specimens shall be conditioned to moisture equilibrium in a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1941 to 1944.

<sup>2a</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

plus or minus 2 F. (1.1 C.) in temperature.

NOTE.—For purpose of tests, it is recognized that specimens reach moisture equilibrium after exposure for at least 4 hr. at the above specified relative humidity and temperature.

### Physical Properties

4. The weight, wall thickness, inside diameter, carriers, ends per bobbin, yarn number, and imperfections shall be determined in accordance with Sections 11 to 17, inclusive, of Standard Methods D 354.

### Asbestos Content

5. The asbestos content shall be determined in accordance with Section 9 of the Standard Specifications and Methods of Test for Asbestos Roving for Electrical Purposes (A.S.T.M. Designation: D 375) of the American Society for Testing Materials.<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# STANDARD METHODS OF TESTING AND TOLERANCES FOR WOVEN GLASS FABRICS<sup>1</sup>



A.S.T.M. Designation: D 579 - 44

ADOPTED, 1943; REVISED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 579; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods of testing and tolerances apply to woven continuous filament and staple glass fabrics for electrical purposes (except woven glass tapes).

## Definitions

2. (a) Definitions of terms relating to woven glass fabrics shall be in accordance with the Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123) of the American Society for Testing Materials.<sup>3</sup>

(b) *Standard Condition*.—Standard condition of glass fabric shall be that reached by the fabric when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent

at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

(c) *Moisture Equilibrium*.—It shall be considered that moisture equilibrium is reached when, after free exposure to air in motion, there is no progressive increase in weight. Moisture equilibrium shall be approached from the dry side (not moisture free).

(d) *Tolerances*.—Tolerances are the limits within which the specified characteristics of the fabric must come to constitute a good delivery on contract.

## TOLERANCES

### Thickness

3. (a) The average thickness of the fabric shall not vary from that specified by more than the following:

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1940 to 1943, being revised in 1941 and 1943.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

Nominal Thickness, in.	Permissible Variations, in.	
	Continuous Filament	Staple
0.005 and under.....	±0.0005	.....
Over 0.005 to 0.010...	±0.001	±0.002
Over 0.010 to 0.015...	±0.002	±0.003
Over 0.015 to 0.025...	.....	±0.003

(b) Individual thickness measurements shall not vary from that specified by more than the following:

Nominal Thickness, in.	Permissible Variations, in.	
	Continuous Filament	Staple
0.005 and under....	±0.001	....
Over 0.005 to 0.010.	±0.0015	±0.003
Over 0.010 to 0.015.	±0.0025	±0.0035
Over 0.015 to 0.025.	.....	±0.0035

### Width

4. (a) The width of the fabric measured inside the selvage shall not vary from that specified by more than plus or minus  $\frac{1}{4}$  in.

(b) The width of the selvage at each edge shall not vary from that specified by more than plus or minus  $\frac{1}{16}$  in.

### Weight

5. The average weight of the fabric shall not vary from that specified by more than plus or minus 10 per cent.

### Count

6. (a) *Warp*.—The average count of warp ends per inch shall not vary from that specified by more than plus or minus 2 ends.

(b) *Filling*.—The average count of filling picks per inch shall not vary from that specified by more than plus or minus 2 picks.

### Breaking Strength

7. The breaking strength of the warp and of the filling shall be not less than that specified.

### Glass Content

8. The glass content shall be not less than that specified.

## METHODS OF TESTING

### Methods of Testing

9. All test procedures not covered in the following Sections 10 to 12 shall be

in accordance with Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.<sup>3</sup>

### Thickness

10. (a) The thickness of the fabric shall be determined in accordance with Method A, B, or C of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>4</sup> In case of dispute, Method C shall be used.

(b) The thickness shall be the average of 10 measurements at 10 different places on the sample not less than 12 in. apart, none of which shall be on a selvage edge. The average thickness and the range in thickness shall be recorded.

### Breaking Strength

11. (a) The breaking strength of the fabric shall be determined on a pendulum type tester conforming to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76).<sup>3</sup> The speed of the pulling clamp shall be  $12 \pm \frac{1}{2}$  in. per min.

(b) Tests shall be made by the raveled-strip method in accordance with Section 11 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39).<sup>3</sup> Tests shall be made in the direction of the warp and of the filling. When the specimens break in the clamps of the testing machine, the test results shall be discarded. In order to prevent breakage of the specimen at the clamps, it is recommended that the specimen be prepared by dipping the ends to within  $1\frac{1}{8}$  in. of the center in an impregnant (Note), and be gripped securely in the clamp with approximately  $\frac{1}{16}$  in. of rubber and  $\frac{1}{16}$  in. of felt on each side of the specimen between the jaws.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE.—A recommended impregnant is a mixture of 25 g. of lindol (tricresyl phosphate), 100 g. of methylmethacrylate and 475 g. of toluene, heated in a water bath to 160 F. (71 C.).

(c) At least five breaking strength determinations shall be made and the average of these five tests shall be the breaking strength of the fabric.

#### Glass Content

12. Five test specimens, each weighing not less than 5 g., shall be dried to constant weight in an oven at 105 to 110 C. (220 to 230 F.) and the weights of the dried specimens recorded, weight *A*.

The specimens shall then be placed in a ventilated oven and heated for 1 hr. at a temperature of  $500 \pm 20$  C. ( $932 \pm 36$  F.). After removal from the oven, they shall be cooled in a desiccator to room temperature and then weighed. The weight of the residue shall be multiplied by 100 and divided by the oven-dry weight of the specimen, weight *A*, to obtain the percentage of glass. The average of five determinations shall be the glass content.

NOTE.—This method does not provide for the removal of inorganic material as such materials ordinarily are not present.

# STANDARD METHODS OF TESTING AND TOLERANCES

## FOR

### WOVEN GLASS TAPES<sup>1</sup>



A.S.T.M. Designation: D 580 - 44

ADOPTED, 1943; REVISED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 580; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These methods of testing and tolerances apply to woven continuous filament and staple glass tapes.

#### Definitions

2. (a) Definitions of terms relating to glass tapes shall be in accordance with the Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123) of the American Society for Testing Materials.<sup>3</sup>

(b) *Standard Condition*.—Standard condition of glass tape shall be that reached by the tape when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted

in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

(c) *Moisture Equilibrium*.—It shall be considered that moisture equilibrium is reached when, after free exposure to air in motion, there is no progressive increase in weight. Moisture equilibrium shall be approached from the dry side (not moisture free).

(d) *Tolerances*.—Tolerances are the limits within which the specified characteristics of the tape must come to constitute a good delivery on contract.

#### TOLERANCES

##### Thickness

3. (a) The average thickness of the tape shall not vary from that specified by more than the following:

Nominal Thickness, in.	Permissible Variations, in.	
	Continuous Filament	Staple
0.005 and under.....	±0.0005	.....
Over 0.005 to 0.010...	±0.001	±0.002
Over 0.010 to 0.015...	±0.002	±0.003
Over 0.015 to 0.025...	.....	±0.003

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1940 to 1943, being revised in 1941 and 1943.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

(b) Individual thickness measurements shall not vary from the nominal thickness specified by more than the following:

Nominal Thickness, in.	Permissible Variations, in.	
	Continuous Filament	Staple
0.005 and under...	$\pm 0.001$	.....
Over 0.005 to 0.010.	$\pm 0.0015$	$\pm 0.003$
Over 0.010 to 0.015.	$\pm 0.0025$	$\pm 0.0035$
Over 0.015 to 0.025.	.....	$\pm 0.0035$

### Width

4. The width of the tape shall not vary from that specified by more than the following:

Nominal Width, in.	Permissible Variations, in.	
	Continuous Filament	Staple
$\frac{1}{2}$ .....	$\pm \frac{1}{32}$	$\pm \frac{1}{32}$
Over $\frac{1}{2}$ .....	$\pm \frac{1}{16}$	$\pm \frac{1}{16}$

### Weight

5. The average weight of the tape shall be calculated in yards per pound and in no case shall the yardage be less than that specified.

### Construction

6. (a) *Ends*.—The total number of ends shall not vary from that specified by more than plus or minus one end.

(b) *Picks*.—The number of picks per inch shall not vary from that specified by more than minus 1 pick or plus 2 picks.

### Breaking Strength

7. The average breaking strength of the tape shall be not less than the specified breaking strength.

### Glass Content

8. The glass content shall be not less than that specified.

## METHODS OF TESTING

### Test Conditions

9. The tape shall be brought to a standard condition (Section 2 (b) and (c)) prior to testing and all tests shall

be made under the standard atmospheric conditions.

NOTE.—Moisture equilibrium under these conditions is reached quickly, 1 hr. being considered ample exposure time.

### Sampling Rolls for Test

10. From each shipment of tape at least three rolls shall be selected and the tests prescribed shall be made on sample lengths cut from each roll. Failure of the first samples selected to conform to the requirements of the specifications will necessitate a selection of the same number of rolls for a second test.

### Thickness

11. (a) The thickness of the tape shall be determined in accordance with Method A, B, or C of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>4</sup> In case of dispute, Method C shall be used.

(b) The thickness shall be taken as the average of ten measurements at ten different places on the sample not less than 12 in. apart, none of which shall be on a selvage edge. The average thickness and the range in thickness shall be recorded.

### Width

12. (a) The width of the tape shall be determined by the use of a standard steel scale graduated to read within the degree of accuracy specified for width tolerances in Section 4. The tape shall lie flat on a smooth surface at the time of measurement.

(b) The width of the tape shall be the average of five measurements made at least 1 yd. apart. The average width and the range in width shall be recorded.

### Weight

13. (a) The weight of the tape shall be determined by accurately measuring

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

and weighing samples 2 yd. in length and then calculating the yards per pound.

(b) Not less than three weight determinations shall be made per shipment, such determinations to be made on different rolls from different packages. The average of these three determinations shall be the average weight of the shipment involved.

### Construction

14. (a) *Ends*.—The total number of ends shall be determined for the full width of the tape.

(b) *Picks*.—The number of picks shall be determined in a length of 1 in.

(c) *Number of Tests*.—The number of ends shall be taken as that secured by making one determination per sample. The number of picks shall be taken as the average of three counts made at different places on the sample. An accepted thread counting magnifying glass shall be used in making the counts.

### Breaking Strength

15. (a) The breaking strength of the tape shall be determined on a pendulum type tester conforming to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76).<sup>3</sup> The speed of the pulling clamp shall be  $12 \pm \frac{1}{2}$  in. per min.

(b) Tests shall be made in the direction of the warp only. When the specimens break in the clamps of the testing machine, the test results shall be discarded. In order to prevent breakage of the specimen at the clamps, it is recommended that the specimen be prepared by dipping the ends to within  $1\frac{3}{8}$  in. of the center in an impregnant (Note), and be gripped securely in the clamps with approximately  $\frac{1}{16}$  in. of rubber and  $\frac{1}{16}$  in. of felt on each side of the specimen between the jaws.

NOTE.—A recommended impregnant is a mixture of 25 g. of lindol (tricresyl phosphate),

100 g. of methylmethacrylate, and 475 g. of toluene, heated in a water bath to 160 F. (71 C.).

(c) Tapes  $1\frac{1}{2}$  in. and under in width shall be tested in full width, and the distance between the clamps shall be 3 in.

(d) Tapes over  $1\frac{1}{2}$  in. in width shall be tested by the raveled-strip method in accordance with Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.<sup>3</sup>

(e) *Number of Breaking Tests*.—At least five determinations of breaking strength shall be made and the average of these five tests shall be the breaking strength of the roll.

### Glass Content

16. Five test specimens, each weighing not less than 5 g., shall be dried to constant weight in an oven at 105 to 110 C. (220 to 230 F.) and the weights of the dried specimens recorded, weight *A*. The specimens shall then be placed in a ventilated oven and heated for 1 hr. at a temperature of 425 to 500 C. (797 to 932 F.). After removal from the oven, they shall be cooled in a desiccator to room temperature and then weighed. The weight of the residue shall be multiplied by 100 and divided by the oven-dry weight of the specimen, weight *A*, to obtain the percentage of glass. The average of five determinations shall be the glass content.

NOTE.—This method does not provide for the removal of inorganic material as such materials ordinarily are not present.

### Sizing Content

17. The average loss in weight after heating at 425 to 500 C. (Section 16) shall be calculated as a percentage of the oven-dry weight, weight *A*, and reported as sizing content.



# STANDARD METHODS OF TESTING AND TOLERANCES

## FOR

### WOVEN GLASS TUBULAR SLEEVING AND BRAIDS<sup>1</sup>



A.S.T.M. Designation: D 581 - 44

ADOPTED, 1943; REVISED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 581; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These methods of testing and tolerances apply to continuous filament and staple glass tubular sleeving and braid.

#### Definitions

2. (a) Definitions of terms relating to glass sleeving and braid shall be in accordance with the Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123) of the American Society for Testing Materials.<sup>3</sup>

(b) *Standard Condition*.—Standard condition of glass sleeving and braid shall be that reached by the fabric when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C) in temperature.

(c) *Moisture Equilibrium*.—It shall

be considered that moisture equilibrium is reached when after free exposure to air in motion, there is no progressive increase in weight. Moisture equilibrium shall be approached from the dry side (not moisture free).

(d) *Tolerances*.—Tolerances are the limits within which the specified characteristics of the textile must come to constitute a good delivery on contract.

#### TOLERANCES

##### Inside Diameter

3. The inside diameter of the glass sleeving and braid shall not be less than that specified and shall not exceed the specified inside diameter by more than the following:

Inside Diameter, in.	Permissible Variations, in.
$\frac{3}{16}$ and under.....	$\pm \frac{1}{64}$
Over $\frac{3}{16}$ to $\frac{1}{2}$ .....	$\pm \frac{1}{32}$
Over $\frac{1}{2}$ .....	$\pm \frac{1}{16}$

##### Wall Thickness

4. The wall thickness of glass sleeving and braid shall not vary from that specified by more than the following:

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1940 to 1943.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

Type of Sleeve or Braid	Permissible Variations in Wall Thickness, in.
Continuous filament.....	$\pm 0.001$
Staple.....	$\pm 0.002$

### Weight

5. The weight of the glass sleeving and braid shall be calculated in yards per pound and shall not vary from that specified by more than plus or minus 10 per cent.

### Construction

6. (a) *Carriers*.—The number of carriers shall be not less than that specified.

(b) *Ends per Carrier*.—The number of ends on the carrier shall not exceed that specified.

(c) *Picks*.—The number of picks per inch shall not vary from that specified by more than plus or minus 3 picks.

### Yarn Number

7. The yarn number shall not vary from that specified by more than plus or minus 10 per cent.

### Glass Content

8. The glass content shall be not less than that specified.

## METHODS OF TESTING

### Diameter, Thickness, Ends

9. The inside diameter, wall thickness, and number of ends per carrier shall be determined in accordance with the Standard Methods of Testing and Tolerances for Tubular Sleeving and Braids (A.S.T.M. Designation: D 354) of the American Society for Testing Materials.<sup>3</sup>

### Picks per Inch

10. The number of picks per inch shall be measured over a 3-in. length after the sleeving or braid has been placed upon a mandrel having the same

diameter as the specified inside diameter of the sleeving or braid. A standard pick counter shall be used in counting the number of picks per inch. The average of three measurements shall be taken as the number of picks per inch.

### Yards per Pound

11. The sleeving or braid shall be slipped over a mandrel approximately 1 yd. in length, the diameter of which corresponds to the specified inside diameter of the sleeving or braid. The material shall be made to lie smoothly on the mandrel. A specimen 2 ft. in length shall be accurately measured from the center of this piece and cut off with a sharp instrument. This specimen shall then be weighed on an analytical balance or other scale of equal accuracy and the yards per pound calculated. The average value obtained from tests on five specimens shall be taken as the yards per pound.

### Glass Content

12. Five test specimens, each weighing not less than 5 g., shall be dried to constant weight in an oven at 105 to 110 C. (220 to 230 F.) and the weights of the dried specimens recorded, weight *A*. The specimens shall then be placed in a ventilated oven and heated for 1 hr. at a temperature of  $500 \pm 20$  C. ( $932 \pm 36$  F.). After removal from the oven, they shall be cooled in a desiccator to room temperature and then weighed. The weight of the residue shall be multiplied by 100 and divided by the oven-dry weight of the specimen, weight *A*, to obtain the percentage of glass. The average of five determinations shall be the glass content.

NOTE.—This method does not provide for the removal of inorganic material as such materials ordinarily are not present.

# STANDARD METHODS OF TESTING AND TOLERANCES

## FOR

### WOVEN TAPES<sup>1</sup>



A.S.T.M. Designation: D 259 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 259; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These methods of testing and tolerances apply to silk and cotton tapes.

#### TOLERANCES

#### Tolerances

2. Tolerances are the limits within which the specified characteristics of the textile must come to constitute a good delivery on contract.

#### Weight

3. The average weight of the tape shall be calculated in yards per pound and in no case shall the yardage be less than that specified.

#### Width

4. The width of the tape shall not vary from that specified by more than the following:

Nominal Width, in.	Permissible Variations, in.
$\frac{1}{2}$ .....	$\pm \frac{1}{32}$
$\frac{3}{4}$ .....	$\pm \frac{3}{32}$
1.....	$\pm \frac{1}{8}$
$1\frac{1}{4}$ .....	$\pm \frac{3}{16}$
$1\frac{1}{2}$ .....	$\pm \frac{1}{4}$
Over $1\frac{1}{2}$ .....	$\pm \frac{5}{16}$

#### Thickness

5. (a) The average thickness of the tape shall not vary from that specified by more than the following:

Nominal Thickness, in.	Permissible Variations, in.
0.011 and under.....	$\pm 0.001$
Over 0.011 to 0.020, incl.....	$\pm 0.0015$

(b) Individual thickness measurements shall not vary from the nominal thickness specified by more than the following:

Nominal Thickness, in.	Permissible Variations, in.
0.011 and under.....	$\pm 0.0015$
Over 0.011 to 0.020, incl.....	$\pm 0.002$

#### Construction

6. The total number of ends shall be not less than specified. The picks per

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1926 to 1927. They were adopted in 1927, published as standard from 1927 to 1933, but revised, and republished as tentative from 1933 to 1944, being revised in 1936, 1939, and 1942.

inch shall not vary by more than the following:

Tape Count	Permissible Variations
48 per inch and under.....	$\pm 1$ pick
Over 48 per inch.....	$\pm 2$ picks

## METHODS OF TESTING

### Sampling Rolls for Test

7. From each shipment of tape at least three rolls shall be selected and the tests prescribed shall be made on sample lengths cut from each roll. Failure of the first samples selected to conform to the requirements of the specifications will necessitate the selection of the same number of rolls for a second test.

### Weight

8. (a) The weight of the tape shall be determined by accurately measuring and weighing samples 2 yd. in length and then calculating the yards per pound.

(b) Not less than three determinations of weight shall be made per shipment, such determinations to be made on different rolls from different packages. The average of these three determinations shall indicate the average weight of the shipment involved.

### Width

9. (a) The width of the tape shall be determined by the use of a standard steel scale graduated to read within the degree of accuracy specified for width tolerances in Section 4. The tape shall lie flat on a smooth surface at the time of measurement.

(b) At least ten measurements of width shall be made on the samples used for the weight determination (Section 8) and the average and range of width recorded.

### Thickness

10. (a) The thickness of the tape shall be determined in accordance with the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.<sup>3</sup>

(b) At least ten measurements of thickness shall be made on the samples used for the weight determination (Section 8) and the average and range of thickness recorded.

### Construction

11. (a) *Ends*.—The total number of ends shall be determined for the full width of the tape.

(b) *Picks*.—The number of picks shall be determined in a length of 1 in.

(c) *Number of Tests*.—The number of ends shall be taken as that secured by making one determination per sample. The number of picks shall be taken as the average of three counts made at different places on the sample. An accepted thread counting magnifying glass shall be used in making the counts.

### Breaking Strength

12. (a) The breaking strength of the tape shall be determined on a testing machine conforming to the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76) of the American Society for Testing Materials.<sup>3</sup> Test results, when specimens break in the clamps of the testing machine, shall be discarded.

(b) The specimens in the direction of the warp shall be tested in full section and the initial distance between clamps shall be 3 in.

(c) The specimens in the direction of the filling shall be tested by the grab method in accordance with Section 10 of Standard Methods D 39<sup>3</sup> and the initial distance between the clamps shall be  $\frac{1}{8}$  in.

(d) At least five determinations of the breaking strength shall be made in each direction and the average of these five tests shall indicate the strength of the roll.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

### Sizing Content

13. Total sizing content of the tape shall be determined in accordance with Section 5 of the Tentative Methods of Quantitative Analysis of Textiles (A.S. T.M., Designation: D 629) of the American Society for Testing Materials.<sup>3</sup> Tests shall be made on a composite sample of approximately 5 g.

### Acidity Test Apparatus

14. (a) The acidity or alkalinity of tapes shall be determined on any standard commercial quinhydrone pH indicator having a sensitivity and readability of 0.02 pH. A suitable and approved type of indicator, its preparation and

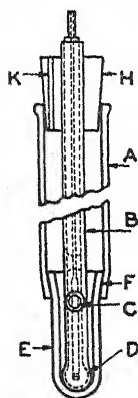


FIG. 1.—Potassium Chloride - Calomel Electrode.

maintenance, are described in the following Paragraphs (b) to (m).

(b) *Potassium Chloride-Calomel Electrode.*—A potassium chloride-calomel electrode assembly is shown in Fig. 1 in which the outside tubes A and E together with the ground glass joint F comprise the salt bridge, while B is the calomel electrode.

(c) *Potassium Chloride.*—A stock solution of potassium chloride shall be prepared by adding special c.p. potassium chloride, free from bromate, to hot

distilled water until saturated and the solution allowed to cool. The solution shall be kept in contact with solid potassium chloride. Before adding to the salt bridge the solution shall be cooled to the lowest temperature at which the test solution will be used. This will prevent crystallization in the salt bridge during use.

(d) The quinhydrone used in the test shall have a quinone content within the limits of 49 to 51 per cent of the total weight.

(e) *Calomel Electrode, Cleaning.*—The calomel electrode, B, shall be cleaned by immersing the lower half in a  $\text{HNO}_3$  solution (1:1) heating the solution gradually to 45 C. (113 F.), and allowing it to cool. The electrode shall then be removed from the acid solution and shall be washed thoroughly with distilled water, and finally with alcohol, and thoroughly dried.

(f) *Calomel Electrode, Filling.*—The small bulb, D, of the calomel electrode shall be half filled with 1 to 1.2 g. of specially purified mercury by introducing it through the hole C by means of a small pipette. Enough mercurous chloride shall be introduced through C to fill the remainder of the bulb D. Two drops of saturated potassium chloride (Paragraph (c)) shall be added to moisten the mixture of mercury and mercurous chloride. A small piece of filter paper shall be held with the finger over the hole C and the electrode shall be vigorously shaken to form an emulsion of the mercurous chloride and mercury globules. The electrode shall be placed in a small beaker containing saturated potassium chloride solution and allowed to stand until the excess calomel has settled into the bulb D. The electrode shall be tapped to remove air bubbles. No air shall be left in the element B

after filling it with saturated potassium chloride solution.

(g) *Salt Bridge*.—Parts *A* and *E* of the salt bridge shall be separately cleaned with chromic acid cleaning solution. The cleaned parts shall be thoroughly washed with distilled water, then with alcohol, and thoroughly dried. The salt bridge shall be assembled by inserting tube *E* into tube *A* as indicated in Fig. 1. Enough saturated potassium chloride solution (Paragraph (c)) shall be added to fill the assembled salt bridge

*B* shall be inserted in the salt bridge as indicated in Fig. 1, adjusting the position of *B* in the cork *H* so that the bulb *D* rests on the bottom of tube *E*. With the bulb *D* resting on the bottom of the tube *E* the level of the potassium chloride solution shall be above the level of the solution being measured when the top of the ground glass joint *F* is at least  $\frac{1}{2}$  in. below the level of the solution being measured.

(i) *Maintenance*.—The following points shall be observed in maintaining

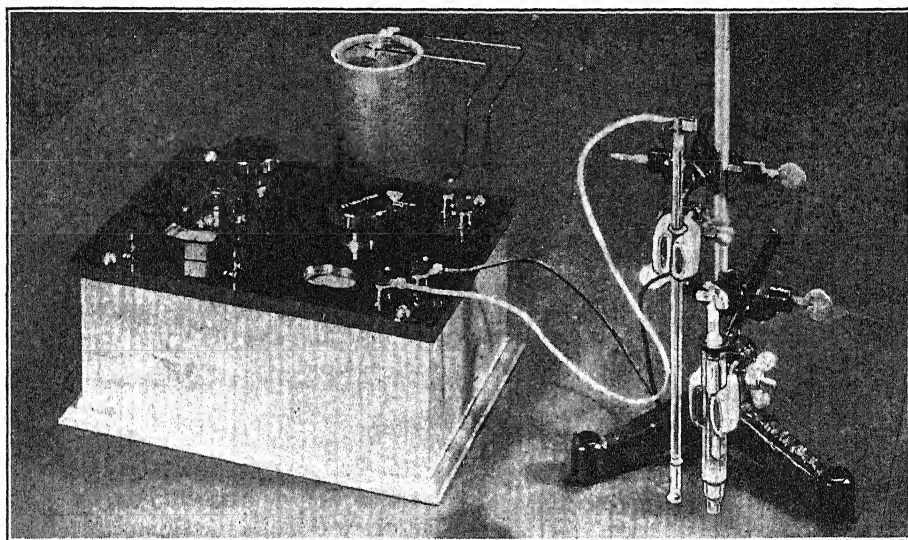


FIG. 2.—Quinhydrone pH Indicator.

approximately one-third full. While holding the tube *A* in a fixed position the tube *E* shall be rotated a slight amount to allow a few drops of the potassium chloride solution to drain out and moisten the ground glass joint *F*. The tube *E* shall be seated gently with a downward twisting motion but not so firmly as to prevent maintaining a moist surface in the joint *F* since the film of potassium chloride solution around the joint *F* forms a part of the electrical circuit.

(h) *Assembly*.—The calomel electrode

the calomel-electrode assembly in satisfactory working condition:

(1) Inspect the breather hole *K* in the stopper *H* daily and see that it is kept open.

(2) Flush the salt bridge daily by turning the tube *E* slightly to allow a few drops of the potassium chloride solution to flow out and reseating *E*, if necessary, as directed in Paragraph (g).

(3) Maintain the level of the potassium chloride solution as directed in Paragraph (h).

(4) If the calomel electrode appears to become insensitive (as is evidenced by the galvanometer pointer swinging free, independent of changes in pH), flush the salt bridge as directed in (2) above.

(j) *Electrode, Platinum or Gold.*—A platinum or gold electrode for use with quinhydrone shall be prepared by fusing a piece of thin platinum or gold ribbon, about 1 sq. cm. in area, through the end of a glass tube. The glass tube shall be filled with ordinary mercury for contact with the wire lead.

(k) *Potentiometer.*—The potentiometer shall be readable to at least 0.001 v.

(l) *Galvanometer.*—The galvanometer shall be sensitive to at least 0.001 v.

(m) *Standard Cell and Battery.*—A Weston or similar standard cell and a 2-v. battery will be required.

#### Acidity Test Procedure

15. (a) *Apparatus Assembly.*—Figure 2 illustrates the apparatus set up showing the two electrodes on the right connected with potentiometric equipment and standard dry cell.

(b) The solution for the acidity test shall be prepared by placing a 10-g. sample of the tape cut into pieces  $\frac{1}{4}$  in. square in a Soxhlet extractor and adding sufficient water to cover it. The extraction shall be carried on for a period of 2 hr. The solution thus obtained shall be transferred to a flask and diluted with recently boiled and cooled distilled water to give a volume of 250 ml. The whole or any portion of this solution may be used for making the pH determination.

(c) The potassium chloride-calomel electrode shall be inserted into the prepared test solution (Paragraph (b)) so that the end of the electrode extends well

below the level of the solution. The platinum or gold electrode shall be inserted into the solution at the opposite side of the calomel electrode. It shall be so placed that the gold or platinum of the electrode is well below the level of the solution. Solid quinhydrone shall be added to the test sample until no more will dissolve. Since quinhydrone is not very soluble, only a small amount will be required. The electrodes, potentiometer, galvanometer, Weston or similar standard cell, and the 2-v. battery shall be connected so as to measure the voltage between the electrodes (Fig. 2). The potentiometer and temperature readings shall be taken 2 min. after the immersion of the electrodes in the sample. The pH value shall be determined from standard tables. During the whole procedure care shall be taken to prevent contamination of the test solution by gases which may be present in the surrounding atmosphere. Duplicate tests shall be made on composite samples of the tape and the pH value reported for each sample.

#### Failure

16. Failure of any of the tape to conform to the requirements of the specifications will make the shipment the subject of complaint and disposition in accordance with the agreement between the purchaser and the seller.

#### Referee Conditions

17. In case of dispute, tests shall be made on material under standard conditions as defined in Section 2 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.<sup>3</sup>



# *Standard Specifications for*

## CAST METHACRYLATE PLASTIC SHEETS, RODS, TUBES, AND SHAPES<sup>1</sup>



A.S.T.M. Designation: D 702 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 702; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These specifications cover methacrylate plastic sheets, rods, tubes, and other shapes produced by casting or machining cast blanks. These specifications do not apply to heat-formed and molded parts and shapes, or sections that are made by assembling two or more pieces.

NOTE.—The properties included in these specifications are those required to identify the types and grades of materials covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Types and Grades

2. (a) These specifications cover the

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1943 to 1946.

following two types of cast methacrylate plastics:

*Type I.*—General purpose material having a heat distortion temperature between 140 and 185 F.

*Type II.*—Heat-resistant material having a heat distortion temperature over 185 F.

(b) These specifications cover the following two grades of material:

*Grade 1.*—Finished surface, having a highly polished surface.

*Grade 2.*—Unfinished surface, having a frosted, sanded, or otherwise unpolished surface.

### General Requirements

3. The size, shape, dimensional tolerances, surface finish, and color shall be as specified by the purchaser in the contract or order (Note).

NOTE.—<sup>2a</sup> Sheet material is available in the standard sizes shown in Table I and is subject to the permissible variations from the specified dimensions shown in Table I.

<sup>2a</sup> Editorially revised in March, 1947.



TABLE I.—STANDARD SIZES AND PERMISSIBLE VARIATIONS FOR CAST METHACRYLATE PLASTIC SHEETS.

Thickness			Width and Length, in. <sup>c</sup>
Nominal, <sup>a</sup> in.	Permissible Variations <sup>b</sup>		
	Under	Over	
0.060.....	0.010 in.	0.010 in.	6 by 12
0.080.....	0.010 in.	0.010 in.	12 by 12
0.100.....	0.012 in.	0.012 in.	12 by 18
0.125.....	0.015 in.	0.015 in.	12 by 24
0.150.....			12 by 36
0.187.....			12 by 48
0.220.....			12 by 60
0.250.....	10 per cent	10 per cent	18 by 18
0.312.....			18 by 24
0.375.....			18 by 36
0.500.....			18 by 60
			20 by 50
			24 by 24
			24 by 36
			24 by 48
			24 by 60
			30 by 36
			36 by 36
			36 by 48
			36 by 60
			40 by 50
0.625.....			6 by 12
0.750.....			12 by 12
0.875.....			12 by 24
1.000.....			12 by 36
1.125.....			12 by 48
1.250.....	10 per cent	10 per cent	20 by 50
1.375.....			24 by 24
1.500.....			24 by 36
1.625.....			24 by 48
1.750.....			36 by 36
1.875.....			36 by 48
2.000.....			40 by 50

<sup>a</sup> Sheet material of thicknesses between 0.060 and 3.000 in. other than those listed in Table I is sometimes available from specific manufacturers.

<sup>b</sup> The permissible variations in thickness apply at any point on the surface of the sheet.

<sup>c</sup> Sheet material is subject to permissible variations of plus or minus  $\frac{1}{16}$  in. in width and length.

## Detail Requirements

4. The materials shall conform to the requirements prescribed in Table II, with the exceptions noted under the column on remarks.

## Sampling

5. A sample shall be selected from sheet  $\frac{1}{8}$  in. in thickness sufficient to determine conformance of the material to these specifications. In the case of materials that are normally processed only into special sizes and shapes from which the required test specimens cannot be cut, the test specimens shall be prepared specially from the material under conditions simulating those of the manufacture of the special size or shape.

## Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—For those tests where conditioning is required, the test specimens of methacrylate plastics shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating

TABLE II.—DETAIL REQUIREMENTS FOR CAST METHACRYLATE PLASTICS.

	Type I, General Purpose	Type II, Heat Resistant	Remarks
Index of refraction, $n_D$ {min.....	1.48	1.49	Clear material only.
{max.....	1.50	1.51	
Specific gravity, 25/25 C. (77/77 F.) {min.....	1.18	1.18	Unpigmented material only.
{max.....	1.20	1.20	
Light transmission, min., per cent.....	91	91	Grade 1, clear, colorless, material only.
Haze, max., per cent.....	3.0	3.0	Grade 1, clear material only—not applicable to sheets over $\frac{1}{2}$ in. in thickness.
Displacement factor (optical), max.....	25 <sup>a</sup>	25 <sup>a</sup>	Grade 1, flat sheets only.
Water absorption:			
Gain in weight, max. per cent.....	0.4	0.4	Material $\frac{1}{8}$ in. in thickness only.
Soluble matter loss, max., per cent.....	0.1	0.1	
Shrinkage, max., per cent.....	1.0	1.0	Sheets only.
Heat distortion temperature {min.....	60 C. (140 F.)	85 C. (185 F.)	
{max.....	85 C. (185 F.)	.....	
at 264 psi. fiberstress	5800	6500	
Tensile strength, min., psi.....	2	2	
Elongation at rupture, min., per cent.....	0.4	0.4	
Impact strength (Izod), min., ft.-lb. per in. of notch.....			

<sup>a</sup> A displacement factor of 25 is equivalent to 7 min. of arc or  $\frac{1}{8}$  in. image displacement at 25 ft.

<sup>b</sup> The light transmission characteristics of colored materials shall be agreed upon by the manufacturer and the purchaser, either on the basis of a visual matching of standard samples or by spectrophotometric means.

Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions*.—The tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(c) *Index of Refraction*.—Standard Methods of Test for Index of Refraction of Transparent Organic Plastics (A.S.T.M. Designation: D 542),<sup>3</sup> using one of the exposed surfaces of the test specimen that has been given any necessary polish without gross removal of material.

(d) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Light Transmission*.<sup>4</sup>—On grade 1, clear, colorless material, measure the light transmission in accordance with the Tentative Method of Test for Luminous Reflectance and Transmission Characteristics and Color of Plastic Materials (A.S.T.M. Designation: D 791).<sup>3</sup> It is recommended that measurements be made with a calibrated photoelectric device:

(f) *Haze*.—Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell (A.S.T.M. Designation: D 672),<sup>3</sup> except that the test specimens shall have a thickness not over  $\frac{1}{2}$  in. Haze shall be measured on grade 1 clear material only.

(g) *Displacement Factor*.—Standard Method of Test for Surface Irregularities of Flat Transparent Plastic Sheet (A.S.T.M. Designation: D 637),<sup>3</sup> using test specimens consisting of flat sheets 18 by 18 in. Measurements shall be made on grade 1 sheets only.

(h) *Water Absorption*.—Standard

Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion procedure and test specimens  $\frac{1}{2}$  in. in thickness. The test specimens shall be conditioned and reconditioned for 24 hr. at  $50 \pm 3$  C. ( $122 \pm 5$  F.).

(i) *Shrinkage*.—The procedure, for sheets only, shall be as follows: On the test specimen 12 by 12 in. mark two fine lines at right angles to each other entirely across the specimen from the mid-points of opposite sides. Across each of these lines place fine gage marks 2 in. in from each edge of the specimen. Measure and record the distance between these gage marks to the nearest 0.01 in. Suspend the specimen from one edge in a circulating air oven, and heat at the recommended forming temperature for 30 min. Remove the specimen from the oven and allow to cool to room temperature while in the suspended position. Remeasure the distance between the gage marks and calculate the shrinkage as the percentage change in distance between the gage marks based upon the original distance. With suitable modifications of technique, other forms besides sheets may also be tested by this method.

(j) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648).<sup>3</sup> The test specimens shall be taken from material as received. Any square, rectangular, or cylindrical test specimen may be used, but the test load used shall be calculated on the basis of a center-loaded beam in which the outer fiber stress produced is 264 psi. Pieces of thin sheets  $\frac{1}{2}$  in. in width may be stacked and bound together to form a test specimen approximately  $\frac{1}{2}$  by  $\frac{1}{2}$  in. If such a specimen is used, it shall be loaded parallel to the width of the assembled pieces.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> Editorially revised in January, 1947.

(k) *Tensile Strength and Elongation at Rupture*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup> The test specimens shall be taken from material as received and determinations made on at least five specimens and the results averaged.

(l) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup> The test specimen shall be taken from material as received.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to the following:

##### *For Sheets:*

Thickness and dimensions,  
Appearance,  
Displacement Factor, and  
Distortion Distance.

##### *For Rods and Tubes:*

All dimensions, and  
Appearance.

The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser. Any material which it is claimed has failed to conform to the requirements of these specifications shall be retained in the original packages until proper disposition has been agreed upon by the manufacturer and the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, form, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Standard Specifications for*

## UREA-FORMALDEHYDE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 705 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 705; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These specifications cover thermo-setting molding compounds consisting of a urea-formaldehyde resin binder that has been intimately combined in the uncured or partially cured condition with fillers, pigments, and other chemical agents.

NOTE.—The properties included in these specifications are those required to identify the types of molding compounds covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Type and Grades

2. These specifications cover a type of urea-formaldehyde molding compound available in the following three grades:

*Grade 1.*—A general purpose molding compound with alpha cellulose filler.

*Grade 2.*—A general purpose molding compound with cellulose filler other than alpha cellulose.

*Grade 3.*—An arc-resistant molding compound with cellulose filler.

### General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The apparent density, bulk factor, flow, particle size, and color shall be as specified by the purchaser in the contract or order.

### Detail Requirements

4. Test specimens molded by compression under conditions specified by the manufacturer shall conform to the requirements as to physical and electrical properties prescribed in Table I.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1943 to 1946.

procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

TABLE I.—DETAIL REQUIREMENTS FOR COMPRESSION MOLDED TEST SPECIMENS.

	Grade 1	Grade 2	Grade 3
Specific gravity, $\frac{1}{\text{min.}}$ .....	1.45	1.45	1.45
25/25 C. (77/77 F.) $\frac{1}{\text{max.}}$ .....	1.55	1.55	1.55
Flexural strength, min., psi. ..	10 000	10 000	10 000
Impact strength (Izod), min., ft.-lb. per in. of notch.....	0.20	0.20	0.20
Water absorption (24-hr. im- mersion), max. weight gain, per cent.....	3.0	3.0	3.0
Dielectric strength, min., $\frac{1}{\text{short-time}}$ .....	300	300	300
v. per mil. .... $\frac{1}{\text{step-by-step}}$ .....	250	250	250
Arc resistance, min., sec.....	...	...	90

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—For those tests where conditioning is required, the molded test specimens of urea-formaldehyde shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(c) *Specific Gravity.*—Method A of the Tentative Methods of Test for Specific

Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(d) *Flexural Strength.*—Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48).<sup>3</sup>

(e) *Impact Strength (Izod).*—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256),<sup>3</sup> except that the test specimen shall be built up of four pieces each  $\frac{1}{8}$  by  $\frac{1}{2}$  in. in cross-section.

(f) *Water Absorption.*—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion.

(g) *Dielectric Strength.*—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> except that the test specimens shall be compression molded disks or plates  $\frac{1}{8}$  in. in thickness, conditioned for 48 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.) at  $50 \pm 2$  per cent relative humidity, and tested under the same conditions.

(h) *Arc Resistance.*—Standard Method of Test for Arc Resistance of Solid Electrical Insulating Materials (A.S.T.M. Designation: D 495).<sup>3</sup>

### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The pur-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

chaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of the two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications

shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, grade, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Standard Method of Test for*

## RESISTANCE OF PLASTICS TO CHEMICAL REAGENTS<sup>1</sup>



A.S.T.M. Designation: D 543 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 543; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method is intended for the testing of all organic plastic materials, including cast, hot-molded, cold-molded, laminated resinous products, and sheet materials, for resistance to chemical reagents. It includes provisions for reporting changes in weight, dimensions, and appearance, but does not cover changes in strength characteristics, electrical properties, and the like. It is intended that the effect of chemicals on these latter properties shall be determined by making measurements on the standard specimens for such tests before and after immersion in the reagents listed herein. In using this method to evaluate the resistance of plastics to chemical reagents, it is recommended that the impact strength of the plastics before and after immersion in the selected reagents also be determined and reported in accordance with the Tentative Methods of Test for Impact

Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256) of the American Society for Testing Materials.<sup>3</sup>

NOTE.—The limitations of the results obtained from this test should be recognized. The choice of types and concentrations of reagents, duration of immersion, temperature of the test, and properties to be reported upon is necessarily arbitrary and serves primarily as a guide to investigators wishing to compare the relative resistance to chemicals of various plastics. For applications involving continuous immersion, the data obtained in a short-time test are of interest only in eliminating the most unsuitable materials. Those with special problems in the application of plastics under corrosive conditions must fit to their special needs the particular compounds and concentrations employed, the duration of contact, the temperature of the system, the physical measurements made, and other experimental factors involved in determining the serviceability of the plastics.

### Apparatus

2. The apparatus shall consist of an accurate chemical balance, containers for test specimens, and a cabinet for maintaining a temperature between 77 F. (25 C.) and 86 F. (30 C.).

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1943, being revised in 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Standard Reagents<sup>4</sup>

3. (a) *Sulfuric Acid (30 per cent)*.—Slowly add 199 ml. (366 g.) of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) to 853 ml. of water.

(b) *Sulfuric Acid (3 per cent)*.—Slowly add 16.6 ml. (30.6 g.) of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) to 988 ml. of water.

(c) *Sodium Hydroxide Solution (10 per cent)*.—Dissolve 111 g. of NaOH in 998 ml. of water.

(d) *Sodium Hydroxide Solution (1 per cent)*.—Dissolve 10.1 g. of NaOH in 999 ml. of water.

(e) *Ethyl Alcohol (95 per cent)*.—Undenatured ethyl alcohol.

(f) *Ethyl Alcohol (50 per cent)*.—Add 598 ml. (482 g.) of 95 per cent undenatured ethyl alcohol to 434 ml. of water.

(g) *Acetone*.

(h) *Ethyl Acetate, c.p.*

(i) *Ethylene Dichloride*.

(j) *Carbon Tetrachloride, c.p.*

(k) *Toluene*.

(l) *Heptane*.—Commercial grade, boiling range 90 to 100 C.

(m) *Sodium Chloride Solution (10 per cent)*.—Add 107 g. of NaCl to 964 ml. of water.

(n) *Distilled Water*.—Freshly prepared.

### Supplementary Reagents<sup>4</sup>

4. (a) *Nitric Acid (10 per cent)*.—Add 108 ml. (153 g.) of  $\text{HNO}_3$  (sp. gr. 1.42) to 901 ml. of water.

(b) *Hydrochloric Acid (10 per cent)*.—Add 239 ml. (283 g.) of HCl (sp. gr. 1.19) to 764 ml. of water.

(c) *Acetic Acid (5 per cent)*.—Add 48 ml. (50.5 g.) of glacial acetic acid (sp. gr. 1.05) to 955 ml. of water.

(d) *Oleic Acid, c.p.*

(e) *Ammonium Hydroxide (10 per*

*cent)*.—Add 375 ml. (336 g.) of  $\text{NH}_4\text{OH}$  (sp. gr. 0.90) to 622 ml. of water.

(f) *Sodium Carbonate Solution (2 per cent)*.—Add 55 g. of  $\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$  to 964 ml. of water.

(g) *Hydrogen Peroxide Solution (3 per cent, or U.S.P. 10 volume)*.—Add 98 ml. (108 g.) of commercial grade (100 volume or 28 per cent) hydrogen peroxide to 901 ml. of water.

### Test Specimens

5. (a) The test specimen for molded materials shall be in the form of a disk 2 in. in diameter and  $\frac{1}{8}$  in. in thickness when comparison with values for other plastics is desired. The surface area of this standard disk is 7.068 sq. in.

(b) The test specimen for laminated and sheet materials shall be in the form of a bar 3 by 1 in. by the thickness of the material, which shall preferably be  $\frac{1}{8}$  in. when comparison with values for other plastics is desired. The surface area of the standard bar is 7 sq. in. The permissible variation in thickness of the standard  $\frac{1}{8}$  in. thick specimen used in this test shall be plus or minus 0.008 in., and the permissible variation in total surface area exposed shall be plus or minus 5 per cent. The cut edges of the specimens shall be made smooth by machining or by finishing with No. 0 or finer sandpaper or emery cloth.

### Procedure

6. (a) The plastic shall be tested in all of the standard reagents and any specified supplementary reagents. Three specimens shall be tested in each reagent. Each specimen shall be weighed separately in the as-received condition. The thickness at the center, and the length and width or both diameters shall be measured to the nearest 0.001 in.

<sup>4</sup> Directions for preparation of reagents are for approximately 1 liter quantities. All percentages are by weight.



(b) The specimens shall not be conditioned, but shall be tested in the as-received condition. Each specimen shall be placed in a separate container and totally immersed in approximately 60 ml. of reagent for 7 days at a temperature between 25 and 30 C. The specimen shall be placed on edge in the container so that it is supported at an angle by the bottom and side wall of the container. The reagents shall be stirred every 24 hr. by moderate manual rotation of the container.

(c) The specimens removed from acid, alkali, and miscellaneous solutions shall be washed with running water, wiped with a dry cloth, and weighed without delay in a closed weighing bottle. The thickness at the center, and the length and width or both diameters shall be measured immediately after weighing.

(d) The specimens removed from solvents shall be wiped dry with a cloth. Some specimens may remain tacky due to dissolved material on the surface or solvent absorbed throughout the specimen. This condition cannot be avoided. The specimens shall be weighed and measured immediately after wiping, as specified in Paragraph (c).

## Report

7. The report shall include the following information for each specimen tested in all the standard reagents and any specified supplementary reagents:

(1) The initial thickness in inches measured at the center to the nearest 0.001 in.,

(2) The percentage loss or gain in weight during immersion for 7 days (Note 1), calculated to the nearest 0.01 per cent, taking the as-received weight as 100 per cent,

NOTE.—For some samples, absorption of the reagent over the 7-day immersion period is nearly balanced by the removal of soluble constituents from the plastic. This type of behavior is exposed by comparing the original weight of the specimen with its weight when dried for 7 days after immersion. A final weight lower than the original weight indicates removal of soluble constituents.

(3) The percentage increase or decrease in the length and width or diameters, and the thickness during immersion for 7 days, taking the as-received dimensions as 100 per cent, and

(4) The general character of the specimen after immersion.

# Standard Method of Test for DIFFUSION OF LIGHT BY PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 636 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 636; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. (a) This method of test is designed to differentiate the relative ability of plastics to diffuse light and is applicable to materials that scatter incident light to an appreciable extent (diffusion). It is not successfully applicable to the measurement of low ranges of scattering (haze) which are associated with slight departures from optical clarity.

(b) An instrument and procedure are described for measuring the reflective and transmissive diffusion of a beam of light by a flat test specimen of the plastic, the face of which is essentially normal to the axis of the light beam, with numerical means for rating the scattering or diffusion in terms of a theoretically perfect light-scattering surface or medium.

## Apparatus

2. (a) The apparatus shall consist of a goniophotometer, the axes of the active elements of which operate in a horizontal plane that passes through the

center of the test specimen. The active elements shall consist of a light source and optical system so oriented as to illuminate the face of the test specimen at its center, a specimen holder which locates the test specimen normally to the axis of the light beam (except as modified by Paragraph (b)), a photosensitive receptor fixed to a radius arm which has the vertical axis of the face of the specimen as its center of rotation, a galvanometer for indicating the current flow in the receptor circuit, and an enclosure or other means for excluding extraneous light. These units shall be combined into one instrument in accordance with the requirements prescribed in Paragraphs (b) to (g):

(b) *Angle of Illumination of Face of Specimen.*—The axis of the light beam shall meet the center of the face of the specimen normally, except in the horizontal plane, where it shall be displaced 3 deg. in a counterclockwise direction. This angle allows the receptor element to intercept the center of any specularly reflected component from the specimen, without interfering with the beam leaving the source.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1943.

(c) *Angular Spread of Light Beam.*—The light beam shall be suitably controlled so that its divergence does not exceed 6 deg.

(d) *Characteristics of Light Source and Receptor.*—The light source shall be an incandescent tungsten filament operating at a temperature between 2800 and 3000° Kelvin or any other source that approximates closely the color temperature of a tungsten filament at the above temperature. The receptor shall have a spectral sensitivity proportional to the luminosity function of the standard I.C.I. observer. Other equivalent source-receptor combinations may be used.

NOTE 1.—A T-8, 100-w. projection lamp, operating at its rated voltage, combined with a Photox barrier-layer photocell approximates the specified source-receptor combination. Any other combination that will give the same results as the standard is also considered equivalent.

(e) *Receptor Unit.*—The receptor unit shall consist of a movable arm carrying at its outer end the photocell held vertically facing the center. The movable arm shall be of sufficient length so that the photocell may just intercept the center of any specularly reflected component without interfering with the beam leaving the source. The photocell surface shall be covered by a mask containing a vertical slot that subtends 2 deg. at the center of rotation. The receptor unit shall be rotatable about the specimen through two quadrants from an angle of 3 deg. clockwise from normal to the face of the specimen (center of specularly reflected beam) through 174 deg., when the cell will be diametrically opposite the light source.

NOTE 2: *Warning.*—At no time should the photocell be exposed to excessive light intensity. For barrier-layer cells, a safe maximum should be maintained by limiting the current output to 100 microamperes per square inch of exposed photosensitive surface. This limit may be ob-

served by covering the cell opening with a neutral filter when necessary, which should then remain in place for the complete test (determination of reflective or transmissive diffusion, or both). A neutral gray photographic negative is convenient.

(f) *Instrument Enclosure and Baffling.*—The instrument may be contained in its own lighttight enclosure or may be operated in a darkened room with black walls, similar to a photographic darkroom. In the latter case the operator shall wear dark clothing, or stand behind the photocell, or both. The interior of any enclosure and all included instrument parts shall be painted a flat optical black.

While making measurements of diffusion by transmission it is necessary to shield the photocell from scattered light originating in the first, or reflection quadrant by a baffle, not more than  $\frac{1}{16}$  in. in thickness and of suitable height, separating the two quadrants. This baffle shall be of such a length that the photocell just clears the outside end, and may be raised off the instrument base to a sufficient height to allow the receptor arm to pass underneath. Both of these openings shall be covered by a loose flap of black opaque cloth or felt.

At the point where the baffle passes over the center of rotation of the receptor arm, a circular hole shall be cut through, against the back of which the face of the specimen shall be placed. The diameter of the hole shall be 2 in. greater than that of the centered light beam.

(g) *Galvanometer.*—A galvanometer of suitable sensitivity shall be employed in conjunction with the photocell for indicating the magnitude of current flow. The receptor-galvanometer circuit shall be calibrated to obtain a relationship between light incident on the sensitive surface and the corresponding current generated.

NOTE 3.—With a barrier-layer cell, a suitable galvanometer is one having a sensitivity of about 0.05 microamperes per millimeter and a period of about 2 sec. This may be shunted by a resistance to reduce its sensitivity when measuring

peculiar to the physical properties of the plastic to be tested (by compression molding, laminating, injection molding, casting, etc.).

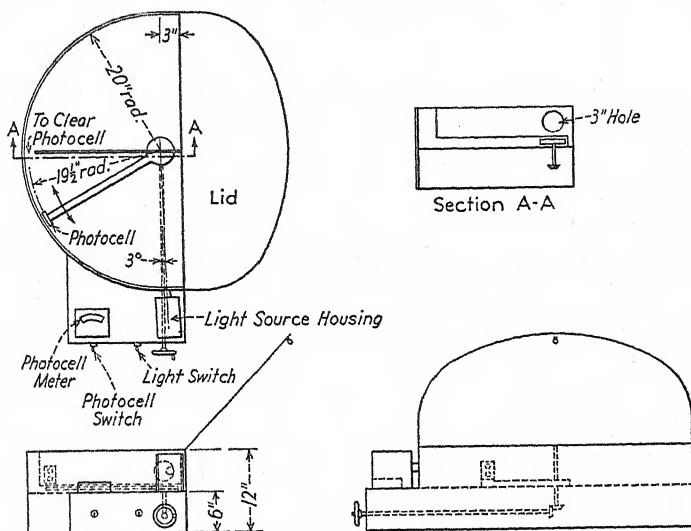


FIG. 1.—Dows-Baumgartner Double Quadrant Reflectometer.

highly reflective or transmissive materials. If a galvanometer of 300 ohms or less internal resistance is employed with the barrier-layer type cell, an essentially linear response is obtained between light flux and current. A calibration should be made in any case, however, with and without any shunt that may be used.

NOTE 4.—Figure 1 shows an instrument devised by Dows and Baumgartner,<sup>3</sup> which has been modified to satisfy the requirements prescribed in Section 2. It is a double-quadrant goniophotometer embodying a light source containing a T-8 100-w. projection lamp, a photocell on a radius arm which is rotated by means of a geared shafting brought out to the front panel, toggle switches, and a galvanometer. The instrument is enclosed in a sheet metal body with an optical black interior. Figure 2 is a photographic view of the interior of the instrument.

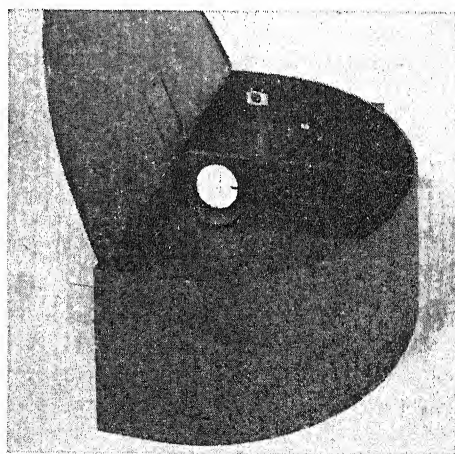


FIG. 2.—Interior View of Dows-Baumgartner Reflectometer.

### Test Specimens

3. (a) The test specimens shall be prepared in the customary manner

(b) The surfaces of the test specimens shall be essentially flat for purposes of measurement, except that, if the surface is configured, the envelope of such configurations shall be flat. If diffusion by transmission is to be measured, the

<sup>3</sup> Dows and Baumgartner, "Two Photovoltaic Cell Photometers for Measurement of Light Distribution," *Transactions, Illum. Engr. Soc.*, Vol. 30, p. 6 (1935).

second surface, or envelope thereof, shall be parallel to the first.

(c) The thickness of the test specimen shall be  $0.050 \pm 0.005$  in. for comparison purposes. If the surface is configured, this restriction shall not hold but the structure and depth of the configurations shall be noted. Other thicknesses may be employed, but only when necessary.

(d) At least ten units of any configured design shall be capable of location within the area of the beam of incident light.

(e) The test specimen shall be sufficiently large to cover completely the specimen hole of the instrument.

### Procedure

4. (a) In making a measurement, the test specimen shall be positioned against the rear surface of the quadrant-separating baffle, covering the hole, and the light turned on. The photocell shall be positioned to give a full-scale or maximum reading on the galvanometer, at or near the specular angle, and any fluctuation or fatigue shall be noted. Fluctuation shall not exceed plus or minus 2.5 per cent of the reading after correcting for nonlinearity of galvanometer response (Section 2 (g)).

NOTE.—Lamp voltage fluctuations may be minimized by employing a voltage-regulating device in the lamp circuit.

(b) Measurements shall be made by either method A or method B. Method A includes the specularly reflected or transmitted component, while method B excludes it. The angles at which the center of the photocell is positioned to take readings for method A shall be as prescribed in Table I; for method B readings of groups 1 and 6 shall be omitted.

(c) At the conclusion of a test by either method, the photocell shall be placed accurately in the starting position

and a reading taken on the galvanometer. This final reading shall check that made at the beginning of the test within plus or minus 2.5 per cent, or the test shall be repeated.

(d) If the specimen is patterned or configured in body or surface texture in a manner that may cause orientation of its diffusive properties, tests shall be

TABLE I.—POSITIONS FOR TAKING BRIGHTNESS READINGS FOR METHOD A (INCLUDING SPECULAR COMPONENT).

Group	Range of Angle of Observation, deg.		Intervals Between Readings, deg.
	From	To	
1 <sup>a</sup> .....	3	15	2
2.....	15	45	5
3.....	45	85	10
4.....	95	135	10
5.....	135	165	5
6 <sup>a</sup> .....	165	177	2

<sup>a</sup> This group of readings shall be excluded for method B.

made with each of the dissimilar axes of symmetry of the pattern in a vertical position and these positions shall be noted.

### Numerical Evaluation of Diffusion

5. (a) The brightness readings shall be plotted as ordinates on Cartesian coordinates, employing the corresponding angle for each reading as the abscissa.

(b) A smooth curve shall be drawn through the points and extrapolated to zero at 90 deg. By means of a planimeter or other sufficiently accurate method, the areas under the brightness-distribution curve, separately for reflective and for transmissive determinations ( $S_r$  and  $S_t$ ), shall be evaluated in any convenient or arbitrary units, from 3 to 90 deg. and from 90 to 177 deg., respectively (method A), or from 15 to 90 deg. and from 90 to 165 deg. (method B).

(c) The area ( $S_{max.}$ ) included under a horizontal straight line at the maximum brightness reading shall be determined over the same angular extent and in the same units of area. This area is the

rectangle that includes the brightness distribution curve for the specimen, within the angular limits corresponding to method A or method B.

(d) The reflective and transmissive diffusion values,  $D_{ra}$  and  $D_{ta}$ , as determined by method A, shall be calculated, as required, by finding the ratio of  $S_{ra}$  and  $S_{ta}$ , respectively, to the corresponding  $S_{max.}$ , as follows:

$$D_{ra} = \frac{S_{ra}}{S_{max.ra}} \dots \dots \dots (1)$$

$$D_{ta} = \frac{S_{ta}}{S_{max.ta}} \dots \dots \dots (2)$$

TABLE II.—TEST RESULTS FOR TYPICAL PLASTIC SPECIMEN 0.052 IN. IN THICKNESS.

Angle of Observation, deg.	Galvanometer Deflection	Angle of Observation, deg.	Galvanometer Deflection
3. ....	8.40 <sup>a</sup>	95. ....	0.01
5. ....	8.40 <sup>a</sup>	105. ....	0.10
7. ....	5.30 <sup>a</sup>	115. ....	0.33
9. ....	3.82 <sup>a</sup>	125. ....	0.52
11. ....	3.28 <sup>a</sup>	135. ....	0.70
13. ....	3.15 <sup>a</sup>	140. ....	0.78
15. ....	3.05	145. ....	0.87
20. ....	2.90	150. ....	0.94
25. ....	2.80	155. ....	1.01
30. ....	2.65	160. ....	1.06
35. ....	2.46	165. ....	1.09
40. ....	2.31	167. ....	1.10 <sup>a</sup>
45. ....	2.10	169. ....	1.10 <sup>a</sup>
55. ....	1.71	171. ....	1.10 <sup>a</sup>
65. ....	1.16	173. ....	1.10 <sup>a</sup>
75. ....	0.61	175. ....	1.10 <sup>a</sup>
85. ....	0.11	177. ....	1.10 <sup>a</sup>

<sup>a</sup> This measurement is used in method A, but not in method B.

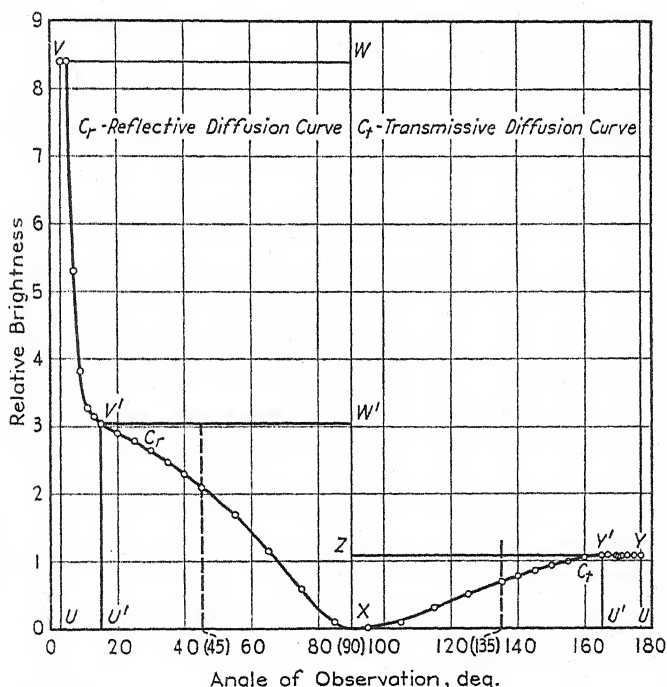


FIG. 3.—Brightness Distribution Curves for Typical Plastic Specimen.

NOTE 1: Example.—Arbitrary brightness readings obtained on a smooth homogeneous plastic specimen 0.052 in. in thickness are given in Table II. The relative brightness curves are plotted in Fig. 3. Curve  $C_r$  is the reflective brightness or diffusion curve, and curve  $C_t$  is the transmissive diffusion curve.

It is seen that:

$D_{ra}$  is evaluated by determining the ratio of the area included within UVX to that included within UVWX, in accordance with Eq. 1).

Likewise  $D_{ta}$  is evaluated by determining the ratio of areas UYX:UYZX;  $D_{rb}$ , by  $U'V'X:U'V'WX$ ;  $D_{tb}$ , by  $U'Y'X:U'Y'ZX$ .

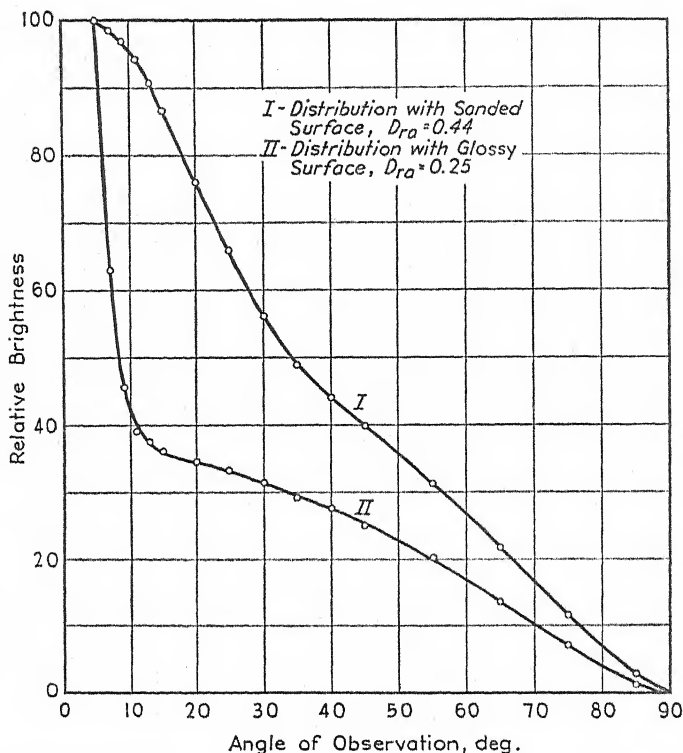


FIG. 4.— Graphical Comparison of Reflective Distribution Curves for Plastic Specimen.

A summation of the areas under the curves gives the following values:

For curve  $C_r$ , by method A, area  $S_{ra} = 91.1$  sq. cm.

For curve  $C_t$ , by method A, area  $S_{ta} = 26.4$  sq. cm.

For curve  $C_r$ , by method B, area  $S_{rb} = 62.1$  sq. cm.

For curve  $C_t$ , by method B, area  $S_{tb} = 19.8$  sq. cm.

The areas under the curve for the perfect diffusor ( $S_{max.}$ ) is as follows:

$S_{max. ra} = 365.4$  sq. cm.

$S_{max. ta} = 47.9$  sq. cm.

$S_{max. rb} = 114.0$  sq. cm.

$S_{max. tb} = 41.3$  sq. cm.

The diffusion values for the material are therefore readily determined as follows:

$$D_{ra} = \frac{S_{ra}}{S_{max. ra}} = \frac{91.1}{365.4} = 0.25, \text{ the reflective diffusing power by method A (including the specularly reflected component).}$$

$$D_{ta} = \frac{S_{ta}}{S_{max. ta}} = \frac{26.4}{47.9} = 0.55, \text{ the transmissive diffusing power by method A.}$$

$$D_{rb} = \frac{S_{rb}}{S_{max. rb}} = \frac{62.1}{114.0} = 0.55, \text{ the reflective diffusing power by method B (excluding the specularly reflected component).}$$

$$D_{tb} = \frac{S_{tb}}{S_{max. tb}} = \frac{19.8}{41.3} = 0.48, \text{ the transmissive diffusing power by method B.}$$

NOTE 2.—It can be seen from the example in Note 1 that the choice of method A or method B depends upon whether or not there is a well defined specularly reflected or transmitted component and also, whether or not it is desired to show the overall diffusing power including this component or to indicate only the light scattering ability of the specimen, independent of its surface characteristics. Obviously, for a material having a matte surface, there is little value in using method A when the simpler method B will give almost the same result. Likewise

method B is to be preferred in transmissive diffusion measurements on a highly diffusive material.

(e) If it is desired to compare graphically the diffusing properties of several specimens by means of their light distribution curves, the maximum brightness reading for each specimen shall be converted to 100 and the readings at other angles shall be modified in like ratio before plotting the points on Cartesian coordinates. The area under each curve is then proportional to the diffusing power of the specimen represented (Fig. 4).

### Report

6. The report shall include the following:

(1) The diffusion values for transmission and reflection, as required, expressed as a decimal,

(2) The values of  $D_r$  and  $D_t$  expressed to the nearest 0.05 above 0.50, to the nearest 0.02 from 0.25 to 0.50, and to the nearest 0.01 below 0.25,

(3) The thickness of the specimen to the nearest 0.001 in., except as noted in Section 3 (c) for special and configured materials, when the necessary dimensions and configurations shall be fully described,

(4) The physical condition of one or both surfaces, depending on whether a determination is made only for  $D_r$ , or whether both  $D_r$  and  $D_t$  are measured, as described by one of the following terms: polished, smooth, matte, grained, pebbled, or configured. If the material is grained, pebbled, or configured, more than one set of results shall be reported, if necessary, orienting the specimen surface differently for each test as specified in Section 4 (d).



# Standard Method of Test for FLAMMABILITY OF PLASTICS 0.050 IN. AND UNDER IN THICKNESS<sup>1</sup>



A.S.T.M. Designation: D 568 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 568; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method of test covers the procedure for determining the flammability of plastics in the form of thin sheets or films 0.050 in. and under in thickness. Two alternate methods of ignition are covered as follows:

*Method A.*—Fusee Method, and

*Method B.*—Benzol Drop Method.

NOTE 1.—For tests of plastics in the form of sheets or plates over 0.050 in. in thickness reference should be made to the Standard Method of Test for Flammability of Plastics Over 0.050 in. in Thickness (A.S.T.M. Designation: D 635) of the American Society for Testing Materials.<sup>3</sup>

## Apparatus

2. The apparatus shall consist of the following:

(a) *Shield.*—A shield constructed from sheet metal or other fire-resistant material, 12 in. in width, 12 in. in depth, and 30 in. in height, and open at the top. The shield shall be so constructed as to provide a ventilating opening

approximately 1 in. in height around the bottom and shall have a viewing window in one side, of sufficient size and in such a position that the entire length of the specimen under test may be observed. Because of danger due to breaking glass, it may be necessary to use heat-resistant glass for the viewing window. One side of the shield shall be hinged (or some other suitable form of construction used) so that the shield may be readily opened and closed to facilitate the mounting and ignition of the test specimen.

(b) *Clamp.*—A spring type of paper clamp for holding the test specimen in position. The holding clamp shall be attached rigidly to the shield in such a manner that when the specimen is clamped therein it is centered within the shield facing the viewing window.

## Test Specimens

3. (a) Not less than six test specimens 1 in. in width by  $12\frac{1}{2}$  in. in length shall be cut from sheets of each of the materials being tested. The thickness of the specimens shall be determined in

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1943, being revised in 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

accordance with method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

(b) The test specimen shall be marked into squares  $\frac{1}{2}$  in. on a side before the test is started. This marking may be done by any convenient means, provided the markings are still visible on the unburned portion of the specimen after the test is completed.

#### *Method A. Fusee Method*

##### **Procedure**

4. (a) *Fusee*.—A pyroxylin plastic fusee material  $0.010 \pm 0.001$  in. in thickness shall be used for ignition. The fusee material shall be made from pyroxylin having a nitrogen content of  $11 \pm 0.10$  per cent and containing 25 per cent camphor. This material shall be cut into 1 in. squares for purpose of tests.

(b) The 1 in. square piece of fusee material shall be affixed by means of acetone to one end of the test specimen, with an overlap of  $\frac{1}{4}$  in. In some cases it may be necessary to dry under slight pressure or to use a stapling device to fasten the fusee to the test specimen. After drying for 2 hr., exposed to the air at  $25 \pm 3$  C., the specimen shall be hung vertically, by means of the supporting clamp (Note 2), in approximately the center of the shield. The fusee shall be at the lower end of the test specimen. The specimen shall be clamped in such a manner that 12 in. of it is exposed below the clamp. The shield shall be placed in a hood, with the ventilating fan turned off at the time of test. The square of fusee material shall be ignited by means of a safety match, and the door of the shield shall be closed immediately (Note 3).

(c) The time required for the flame to either extinguish itself or to completely

burn the test specimen shall be determined by means of a stop watch or timer, started at the instant of ignition of the fusee. The area of the specimen that is burned or charred shall be measured to the nearest  $\frac{1}{4}$  sq. in. The markings on the specimen may be used for estimating the amount of material burned. If material melts and drops from the specimen, the area melted shall be included in the burned area. The charred portion which drops off when touched with the fingers or in handling shall also be included in the burned area.

NOTE 2.—Continued testing of highly flammable materials causes the holding clamp (Section 2 (b)) to become heated. It is recommended that after each determination the holding clamp be cooled to room temperature by means of a cloth soaked in cool tap water.<sup>4</sup>

NOTE 3.—It should be noted that for some materials the products of burning are toxic, and care should be taken to guard the operator from the effects of these toxic gases. The ventilating fan in the hood under which the test is performed should be turned on immediately after the test is completed in order to remove any irritating products of the test.

(d) Another convenient method, which may be used for measuring the burned area in the case of materials that do not stretch under test, is to use a scale 12 in. in length by 1 in. in width ruled with lines into squares  $\frac{1}{2}$  in. on a side. The partially burned test specimen may be laid over this scale, and the amount of burned material estimated by means of the area of the scale left exposed.

#### *Method B. Benzol Drop Method*

##### **Procedure**

5. (a) *Benzol*.—Ordinary laboratory grade benzene ( $C_6H_6$ ) shall be used for ignition.

(b) A drop of benzol shall be placed on the test specimen approximately  $\frac{1}{4}$  in. above the lower edge of the specimen, and allowed to run down to form a large

<sup>4</sup>This note was added editorially in November, 1944.

drop on the blunt end without dripping off. This drop shall be ignited within 7 sec. after application to the test specimen with either of the following means: (1) A high potential, low energy spark such as delivered by an automobile ignition coil, or (2) A safety match. After ignition, the door of the shield shall be closed immediately (Note 3).

(c) The time required for burning and the area burned shall be observed in

accordance with the procedure described in Section 4 (c) and (d) (Note 2).

#### Report

6. The report shall include the following:

- (1) Thickness of the material,
- (2) Length of time in seconds that the material continues to burn,
- (3) Area in square inches of the burned portion of the test specimen, and
- (4) Method of ignition.

*Standard Method of Test for*  
FLAMMABILITY OF PLASTICS OVER 0.050 IN. IN  
THICKNESS<sup>1</sup>



A.S.T.M. Designation: D 635 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 635; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. This method of test covers the procedure for determining the flammability of plastics in the form of sheets or plates over 0.050 in. in thickness.

NOTE.—For tests of plastics in the form of thin sheets or films 0.050 in. and under in thickness reference should be made to the Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568) of the American Society for Testing Materials.<sup>3</sup> Flammability tests of sheet and plate insulation are covered in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials.<sup>3</sup>

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Test Chamber*.—A room or enclosure protected from air currents, but provided with means for venting the fumes from burning specimens. A hood may be used if its exhaust fan is turned

off during the test and allowed to run only periodically to clear out the fumes between tests.

(b) *Ring Stand*.—A laboratory ring stand with two clamps.

(c) *Burner*.—A bunsen burner or an alcohol lamp.

(d) *Wire Gauze*.—A piece of 20-mesh wire gauze, 5 in. square.

(e) *Stop Watch*.

#### Test Specimens

3. (a) At least three test specimens 6 in. in length by 0.5 in. in width and of the thickness of the material as normally supplied shall be cut from each of the materials being tested. The specimens shall be tested in the as-received condition. Special preconditioning may be agreed upon by the purchaser and the seller.

(b) Each test specimen shall be marked by scribing two lines 1 in. and 5 in. from one end of the specimen.

#### Procedure

4. (a) The specimen shall be clamped in a support, at the end farthest from the 1-in. mark, with its longitudinal axis

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard this method was published as tentative from 1941 to 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

horizontal and its transverse axis inclined at 45 deg. to the horizontal. Under the test specimen there shall be clamped a piece of 20-mesh bunsen burner gauze about 5 in. square, in a horizontal position  $\frac{1}{4}$  in. below the edge of the specimen,

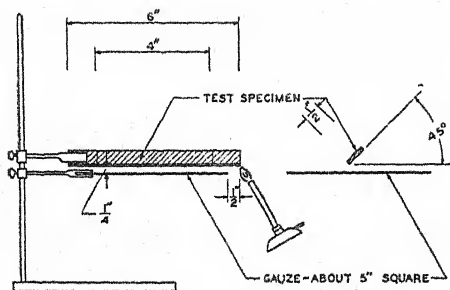


FIG. 1.—Apparatus for Flammability Test.

and with about  $\frac{1}{2}$  in. of the specimen extending beyond the edge of the gauze (see Fig. 1).

(b) A bunsen burner or an alcohol lamp with a flame  $\frac{1}{2}$  to  $\frac{3}{4}$  in. in height shall be placed under the free end of the

test specimen and adjusted so that the flame tip is just in contact with the specimen. At the end of 30 sec. the flame shall be removed and the specimen allowed to burn. A stop watch shall be started when the flame reaches the first mark, 1 in. from the end, and the time observed when the flame reaches the 5-in. mark. In case the plastic does not continue to burn after the first ignition, the burner shall be placed under the free end for a second period of 30 sec. immediately following the extinction of the flame. If the sample does not continue burning to the 5-in. mark after the second ignition, the sample shall be reported as self-extinguishing.

#### Report

5. The report shall include the following:

- (1) The thickness of the material,
- (2) Number of ignitions used, and
- (3) Rate of burning in inches per minute, average of three tests.

# Standard Method of Test for MAR RESISTANCE OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 673 - 44

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 673; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method of test is designed to measure the resistance of glossy surfaces to abrasive action. Since measurement of progressive optical deterioration of a glossy surface is involved, such materials as molded and laminated plastics, paint and lacquer films, and plated coatings may be evaluated by this method. The test consists essentially of two steps as follows:

(1) The production of a series of abraded spots on the surface of the test specimen, made by dropping increasing amounts of abrasive through a tube of fixed length, and

(2) The measurement of the gloss of these abraded spots by an optical method and comparison with the original gloss.

NOTE.—Since this method is intended for rating relative decrease in gloss, obviously only materials having originally high specular reflectance can be measured.

## Apparatus

2. The apparatus shall consist of the following:<sup>3</sup>

(a) *Abrader*.—An abrader, shown in Fig. 1, consisting of the following:

(1) *Tube*.—A glass, metal, or plastic tube (*A*, Fig. 1) supported vertically through which the abrasive is dropped on a test specimen that is supported at an angle of 45 deg. to the axis of the tube. The tube shall conform to the dimensions shown in Fig. 2.

(2) *Hopper*.—A hopper (*B*, Fig. 1) to distribute the feed of the abrasive in order to produce on the specimen an abraded spot nearly uniform in appearance over an area about  $\frac{3}{4}$  in. in diameter. The hopper shall be rotated at about 7 rpm. and the abrasive fed from the hopper at a rate of about 200 to 250 g. per min. The details of construction of the hopper are shown in Fig. 2. It may be formed from thin sheet metal with soldered seams. It shall have a double conical bottom with 6 holes 0.070 in. in diameter arranged in a circle

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

<sup>3</sup> Complete working drawings for this apparatus are obtainable at a nominal charge from the Headquarters of the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

$\frac{5}{8}$  in. in diameter at the bottom edge, and 1 hole 0.070 in. in diameter at the apex of the middle cone. The inside of the hopper should be smooth and highly polished.

(3) *Ball Bearing*.—A ball bearing (C, Fig. 1 and 2)  $2\frac{1}{4}$  in. in outside diameter and  $1\frac{1}{4}$  in. in inside diameter, with sealed balls. The outside ring of the bearing shall be rigidly held in position.

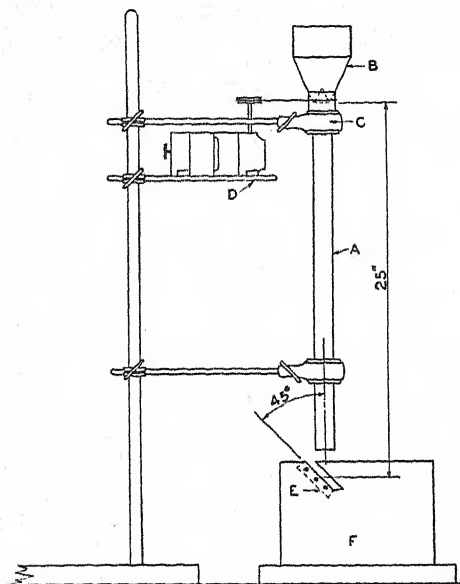


FIG. 1.—Abrader.

(4) *Brass or Copper Tube*.—A brass or copper tube (see Fig. 2) about  $1\frac{1}{2}$  in. in length fitted tightly into the bearing. The inside diameter shall be slightly over 1 in. to permit the top of glass tube A to extend up into it just below the lower edge of the hopper. To the tube shall be soldered 4 supports for holding the hopper in position. The projecting portion of the tube serves as a pulley to rotate the hopper assembly.

(5) *Electric Motor*.—A small electric motor<sup>4</sup> with built-in reducing gears for

rotating the hopper assembly at a speed of about 7 rpm.

(6) *Receptacle for Abrasive*.—A receptacle (F, Fig. 1) about 8 by 8 by 8 in. to receive the abrasive. A 5-gal. can cut down to a height of 8 in. will be satisfactory. The receptacle shall be slotted on two sides at an angle of 45 deg. as shown in Fig. 1 to permit sliding

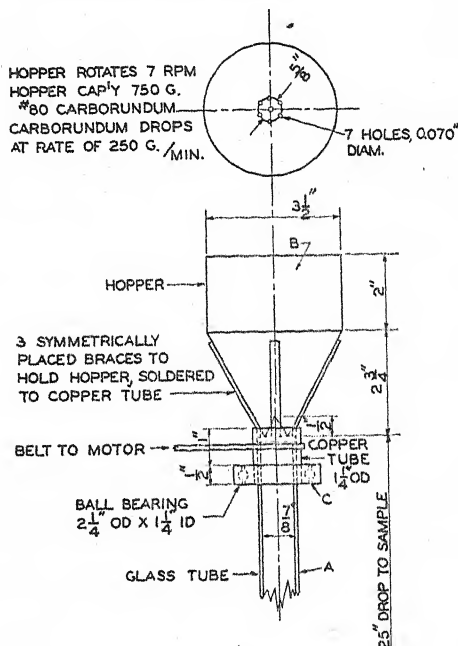


FIG. 2.—Details of Hopper Assembly.

sheet specimens past the bottom of the glass tube.

(7) *Support for Test Specimen*.—A plywood board (E, Fig. 1), with spring clips to hold the test specimen, mounted in the receptacle at an angle of 45 deg. to the axis of the tube and directly under the tube.

(8) *Abrasive*.—No. 80 carborundum or an equivalent abrasive.

NOTE.—It has been found that continued use of the same batch of abrasive gradually improves the values of abrasion resistance of a given material, possibly due to the breaking up of particles of the abrasive or by dulling their sharp cutting edges. An arbitrary limit of 50 tests is recommended for a given lot of abrasive.

<sup>4</sup> A suitable electric motor is the No. 952W/11 rpm. of





may be fastened to a hinged arm with its center in the plane of the test specimen and in line with the center of the illuminated spot, and shall have freedom of motion from the specular position at an angle of 45 deg. to the plane of the specimen to the off-specular position at an angle of 60 deg. to the plane of the specimen. When the glossmeter is assembled and a mirror placed in the position to be occupied by a test specimen, the circular incident light beam from the projector should be seen entering the receptor lens at the specular angle of 45 deg. When the receptor is tilted upward 15 deg. to the off-specular angle of 60 deg., the specular beam shall just miss the receptor lens. The active surface of the photoelectric cell shall be masked to permit only the specularly reflected image of the spot to impinge on the cell.

NOTE.—The glossmeter described may be used to measure gloss on any type of surface, transparent or opaque. For testing of transparent sheets, some form of hazemeter or densitometer may also be used.

### Test Specimen

3. The test specimen shall consist of any material with a flat surface at least 2 by 2 in. in area for each determination. A sheet 2 by 10 in. will be sufficient for five tests.

### Procedure

4. (a) Five abraded spots shall be made on each test specimen (Note), and each succeeding spot shall be produced by using a larger quantity of abrasive than that used for the preceding one. The following amounts of abrasive are recommended:

Spot	Abrasive, g.
No. 1.....	200
No. 2.....	400
No. 3.....	800
No. 4.....	1200
No. 5.....	1600

NOTE.—For a quick test at a mild degree of abrasive action, the results obtained using 200 or 400 g. of abrasive may give sufficient information.

(b) *Abrading Test Specimens.*—The test specimen shall be clipped to the supporting board, the hopper started rotating, and the proper amount of abrasive poured into the hopper. When all of the abrasive has passed through the glass tube, the specimen shall be moved to another position, and the next succeeding amount of abrasive poured into the hopper to produce another

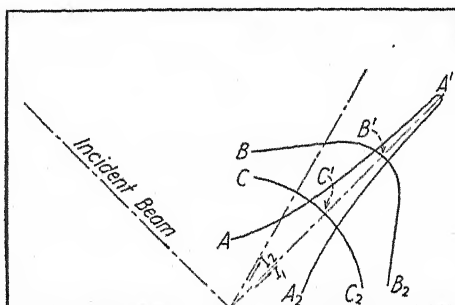


FIG. 4.—Distribution of Reflected Light.

abraded spot. In this manner, five abraded spots shall be produced, each by the respective amount of abrasive. It is recommended that a mild air blast be used to remove excess abrasive adhering to the surface of the specimen after the abraded spots have been produced.

(c) *Measurement of Gloss.*—The gloss of the abraded test spots shall be measured by means of the glossmeter and compared with the original gloss of the test specimen. The principle of operation of the glossmeter in measuring the gloss is based upon the following optical behavior of plane surfaces as shown in Fig. 4. A beam of light incident upon a mirror at an angle of 45 deg. is reflected specularly at 45 deg. and its polar distribution will have the shape of curve  $AA'A_2$ . If the mirror surface is disturbed by abrasion, progressively

more light will be scattered on either side of the specular beam, and the light-distribution curve assumes the shape  $BB'B_2$ , approaching the uniform distribution shown by curve  $CC'C_2$ , which is characteristic of a nearly perfect diffusing surface such as magnesium carbonate. If a lens is placed in the path of the reflected beam and the image of the illuminated spot focused on a photoelectric cell and readings of the intensity taken at the specular angle  $I_1$  and at an angle  $I_2$ , 15 deg. from the specular angle, then the ratio of the readings is a measure of the sharpness of the peak of the light-distribution curve or a measure of the gloss. The angle of 15 deg. off specular is selected because with the prescribed glossmeter the specular beam from a mirror surface just misses the opening of the collecting lens.

NOTE.—For strict comparisons of mar resistance characteristics, materials of nearly similar reflectance values should be used, for example, a comparison of two types of resin coatings in which one was an opaque white and another an opaque black would not be as valid as if both materials had the same general color.

#### Calculating Percentage Gloss

5. (a) To avoid the complication of variability of photoelectric cell readings from surfaces of various original reflectivity and to minimize the effect of voltage variation on the light source, only the ratio of light intensity reflected at the two observation angles shall be used in the following formulas for calculating percentage gloss:

$$\text{Gloss, per cent} = 100 \left( \frac{I_1 - I_2}{I_1} \right)$$

where:

$I_1$  = photoelectric cell reading at the specular angle,

$I_2$  = photoelectric cell reading at the 60 deg. angle (15 deg. off specular), and

$\frac{I_1}{I_2}$  = measure of the sharpness of the peak of light distribution.

From this formula, a reading of zero at the off-specular angle gives 100 per cent gloss, and equal readings at specular and off-specular angles give 0 per cent gloss.

(b) In comparing abrasion characteristics of a series of materials which do not have the same original gloss, the initial gloss value of the unabraded surface shall be taken as 100 per cent, and the progressive deterioration expressed in corresponding terms.

NOTE.—Typical data are shown in the following table:

Abrasive, g.	Readings at:		Gloss, <sup>a</sup> per cent	Percent- age of Original Gloss
	45 Deg., $I_1$	60 Deg., $I_2$		
0.....	93.5	2.0	97.9	100
200.....	60.0	4.0	93.3	95.4
400.....	43.5	5.0	88.5	90.4
800.....	33.0	6.0	81.8	83.5
1200.....	25.0	6.0	74.0	75.5
1600.....	19.0	6.0	68.4	70.0

$$\text{Gloss, per cent} = 100 \left( \frac{I_1 - I_2}{I_1} \right)$$

#### Plotting Results

6. The percentage gloss of the abraded spots shall be plotted against the respective amounts of abrasive used, to obtain a characteristic curve. Since such curves for different materials are often found to change slope irregularly, they may cross each other, and the rating of a series of different materials using a given amount of abrasive may not be representative of their relation at other amounts of abrasive. One way to arrive at an over-all performance is to average the percentage original gloss at the various amounts of abrasive, which would represent the area included between the curve and the coordinate representing the amount of abrasive. (See Note, Section 4, (a))

# *Standard Methods of Test for*

## INDEX OF REFRACTION OF TRANSPARENT ORGANIC PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 542 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 542; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These methods of test are intended for the measurement of the index of refraction of transparent organic plastic materials including cast, hot-molded and sheet materials.

(b) Two procedures, refractometric and microscopic methods, are proposed in order to cover satisfactorily the maximum range of indices found in these materials. The refractometric method is to be preferred wherever it is applicable.

### Temperature Tests

2. All measurements should be made at a temperature of  $25 \pm 5$  C. If a material is found to have a high thermal coefficient of index, the temperature shall be accurately controlled to 25 C.

### REFRACTOMETRIC METHOD

#### Apparatus

3. The apparatus for the preferred method shall consist of an Abbé re-

fractometer (Note 1), a suitable source of white light, and a small quantity of a suitable contacting liquid (Note 2).

NOTE 1.—Other suitable refractometers can be used with appropriate modification of procedure as described in Section 5.

NOTE 2.—A satisfactory contacting liquid is one which will not soften or otherwise attack the surface of the plastic within a period of 2 hr. of contact. The index of refraction of the liquid must be higher, by not less than one unit in the second decimal place, than the index of the plastic being measured.

The following liquids are suggested:

Plastic	Contacting Liquid
Phenol formaldehyde resin.....	$\alpha$ -bromnaphthalene
Cellulose acetate and cellulose nitrate.....	<div style="display: inline-block; vertical-align: middle;"> <div style="display: inline-block; vertical-align: middle;">cassia oil</div> <div style="display: inline-block; vertical-align: middle;">aniseed oil</div> <div style="display: inline-block; vertical-align: middle;"><math>\alpha</math>-bromnaphthalene</div> </div>
Acrylic resins.....	saturated aqueous solution of zinc chloride made slightly acid
Vinyl resins <sup>a</sup> .....	$\alpha$ -monobromnaphthalene
Styrene resins.....	saturated aqueous solution of potassium mercury iodide (Rohrbach's solution). <sup>b</sup>

<sup>a</sup> No liquid has been found to date which is entirely satisfactory for vinyl resins.

<sup>b</sup> Avoid contact of this liquid with the skin or prolonged contact with the metal parts of the refractometer.

NOTE 3.—In many cases a liquid as suggested above is not available and a second rapid reading must be made on a fresh specimen after preliminary trial.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1939 to 1942, being revised in 1940.

### Test Specimen

4. (a) The test specimen shall be of a size such as will conveniently fit on the face of the fixed half of the refractometer prisms (Note 1). A specimen measuring 0.25 in. (6.3 mm.) by 0.5 in. (12.7 mm.) on one face is usually satisfactory.

(b) The surface to be used in contact with the prism shall be flat and shall have a good polish. A second edge surface perpendicular to the first and on one end of the specimen shall be prepared with a fair polish (Note 2). The polished surfaces shall intersect without a beveled or rounded edge.

NOTE 1.—For maximum accuracy in the refractometer method the surface contacting the prism shall be quite flat. This surface can be judged for flatness, provided the specimen has been satisfactorily polished, by observing the sharpness of the dividing line between the light and dark field. A sharp, straight dividing line indicates a satisfactory contact of the specimen and prism surfaces.

NOTE 2.—It has been found possible to prepare a satisfactorily polished surface by hand polishing small specimens on an abrasive material backed by a piece of plate glass. Fine emery paper (for example, No. 000 Behr-Manning polishing paper) followed by a polishing rouge suspended in water on a piece of parchment paper have been used as the abrasive.

### Procedure

5. The hinged illuminating prism shall be removed from the refractometer, if necessary. A source of diffuse light shall be placed so that good illumination is obtained along the plane of the surface of contact between the specimen and the refractometer prism. A small drop of a suitable contacting liquid shall be placed on the polished surface of the specimen and the specimen then placed in firm contact with the surface of the prism and with the polished edge of the specimen toward the source of light. The index of refraction shall be deter-

mined in the same manner as for liquids. This shall be done by moving the index arm of the refractometer until the field seen through the eyepiece is one-half dark. The compensator (Amici prisms) drum shall then be adjusted to remove all color from the field. The index arm shall then be adjusted by means of the vernier until the dividing line between the light and dark portions of the field exactly coincides with the intersection of the cross hairs as seen in the eyepiece. The value of the index of refraction for the sodium D lines shall then be read directly from the instrument. The dispersion shall also be determined by reading the compensator drum and applying this figure, along with the index of refraction, to a chart or table supplied with the instrument.

NOTE.—Sodium light from some type of a sodium burner is of use in increasing the accuracy and ease of setting of the refractometer.

### MICROSCOPIC METHOD

#### Apparatus

6. The apparatus for the auxiliary method shall consist of a microscope having a magnifying power of at least 200 diameters. This microscope shall be equipped with a means of measuring the longitudinal travel of the lens tube to within 0.001 in.

#### Test Specimen

7. The specimen described in Section 4 will be satisfactory for use with the microscopic method if it is not too thick to permit focusing the microscope through the polished surface (having a fair polish). A small specimen taken from a sheet of the material may be used if it is about 0.25 in. (6.3 mm.) in thickness.

### Procedure

8. A specimen having two parallel surfaces shall be placed on the measuring microscope table with that surface having the best polish placed nearest to the objective. The microscope shall then be focused through the specimen and on the bottom surface. The reading of the longitudinal displacement of the lens tube shall be noted to the nearest 0.001 in. or less. Without moving the specimen, the microscope shall then be focused on the top surface of the specimen and the reading of the displacement again noted. The difference between these two readings is the apparent thickness of the specimen. The index of refraction is found by dividing the actual thickness of the specimen by the apparent thickness.

### REPORT

#### Report

9. The report shall include the following:

- (1) The name of the method used.
- (2) The index of refraction to the nearest significant figure warranted by the accuracy and duplicability of the measurement. If the index is specified to more than three significant figures, the wave length of light for which the measurement was made shall be specified.

NOTE.—In the case of nonisotropic materials, for example, injection and compression molded materials, the index observed by the Abbé refractometer method will be the average value for a thin layer of small area at a point of contact near the center of the refractometer prism. For a complete and accurate determination of the variation of the index throughout the test specimen it is necessary to make the measurement at more than one point on the surface and within the body of the material. This can be done by preparing a contacting surface both perpendicular and parallel to the molding pressure or flow. After the test specimen is contacted to the prism it may be translated carefully for short distances along the prism surface in the direction of the light source while the variation of index is followed with the refractometer. This procedure should be repeated a sufficient number of times and for a sufficient number of specimens to determine the range of indices involved. The average value and the range of the index readings obtained from these specimens shall be reported if the range exceeds the accuracy of the measurement. If the variations in index is systematic with the orientation of the test specimen and if these variations exceed those found between specimens of the same material, the nature of these variations shall be reported with the average value.

Care should be taken to work rapidly to avoid changes in the refractive index of the plastic due to its absorption of the contacting liquid.

- (3) The temperature in degrees Centigrade at which the index was measured.
- (4) If available, the dispersion shall be reported along with the index of refraction.

# Standard Method of Test for SHEAR STRENGTH OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 732 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 732; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method covers the punch type of shear test and is intended for use in determining the shear strength of test specimens of organic plastics in the form of sheets and molded disks in thicknesses from 0.005 to 0.500 in.

## Definition

2. *Shear Strength*.—Shear strength is the maximum load required to shear the specimen in such a manner that the moving portion has completely cleared the stationary portion. It is expressed in pounds per square inch based on the area of the sheared edge or edges.

## Apparatus

3. The apparatus shall consist of the following:

(a) *Testing Machine*.—Any suitable testing machine of the constant-rate-of-crosshead speed type. The testing machine shall be equipped with the necessary drive mechanism for impart-

ing to the crosshead a uniform, controlled velocity with respect to the base. The testing machine shall also be equipped with a load-indicating mechanism capable of showing the total compressive load carried by the test specimen. This mechanism shall be essentially free from inertia-lag at the specified rate of testing and shall indicate the load with an accuracy of plus or minus 1 per cent of the indicated value or better. The accuracy of the testing machine shall be verified in accordance with the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials.<sup>3</sup> The machine shall be constructed of such material and in such proportions that the total elastic longitudinal strain of the system does not exceed 1 per cent of the total compressive strain of the test specimen at any time during the test and at any load up to the rated capacity of the machine.

(b) *Shear Tool*.—A shear tool of the punch type which is so constructed that the specimen is rigidly clamped

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1943 to 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials,<sup>3</sup> except that the time of conditioning for materials conditioned in accordance with Functional Procedure A of Methods D 618 shall be limited to 48 hr. regardless of thickness, and materials may be tested, after cooling in a desiccator, as soon as room temperature is reached.

### Procedure

6. (a) Tests on materials conditioned in accordance with Functional Procedure A of Methods D 618 shall be made at room temperature maintained at  $25 \pm 8$  C. ( $77 \pm 15$  F.). Tests on materials conditioned in accordance with the Standard Procedure of Methods D 618 shall be made at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and at a relative humidity of  $50 \pm 2$  per cent.

(b) Five specimens shall be used.

(c) The specimen shall be placed over the  $\frac{3}{8}$ -in. pin of the punch and shall be fastened tightly to it by means of the washer and nut. The tool jig shall then be assembled and the bolts tightened.

(d) The crosshead speed of the machine during test shall be 0.05 in. per min., measured when the machine is running idle.

(e) The punch shall be pushed down

far enough so that the shoulder clears the specimen proper. The specimen will then be adjacent to the necked-down portion of the punch and it should be possible to remove the specimen readily from the tool.

NOTE.—For thick specimens of some materials the punched-out piece tends to stick in the die. If the test is continued only to the point where maximum load has been developed and starts to fall off rapidly, the specimen may be readily removed from both punch and die.

### Report

7. The report shall include the following:

(1) Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, etc.,

(2) Method of test, type of test specimen, and dimensions,

(3) The atmospheric conditions in the test room,

(4) The conditioning procedure used,

(5) Diameter of punch,

(6) The load in pounds required to shear each specimen, and

(7) The calculated shear strength in pounds per square inch determined by dividing the load by the area of the sheared edge, which shall be taken as the product of the thickness of the specimen by the circumference of the punch. Report individual and average values, also the average deviation.



# Standard Method of Test for SPECTRAL CHARACTERISTICS AND COLOR OF OBJECTS AND MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 307 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 307; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method of test is intended to determine the spectral characteristics of light-transmitting objects and of light-reflecting objects and materials, and to designate their colors.

NOTE 1.—The method of designating colors has come into wide acceptance since its recommendation in 1931 by the International Commission on Illumination.<sup>3</sup> It is intended primarily for light-reflecting and light-transmitting specimens whose spectral characteristics have been determined by the spectrophotometer, but it is also applicable to the expression of color measurements by visual or photoelectric colorimeters.

NOTE 2.—If portions of a test specimen to which the measurement applies are homogeneous, the measurement is taken as characteristic of the material of the specimen; thus, a specimen consisting of an opaque paint film or an opaque

layer of paper is a specimen of material (paint or paper). But if the effective portions of the specimen consist of two or more materials so combined that the measurement depends upon their arrangement, the measurement characterizes, not a material, but objects which are constructed from the same materials in the same way; thus, a specimen consisting of an incompletely hiding paint film on a ground coat represents, not a material, but simply a group of objects made from the superimposition of a particular finishing coat on a particular ground coat. A specimen of light-reflecting material shall consist of a layer so thick that further increase in thickness will not cause a change in appearance.

## Description of Terms and Symbols

2. (a) *Tristimulus Designations, X, Y, Z.*—The evaluations of the color as determined in accordance with this method.

NOTE.—The appearance of materials and objects is not completely defined by this evaluation of color, but may also be influenced by other properties such as texture and gloss.

(b) *Tristimulus Designations of a Spectrum of Unit Irradiance per Unit Wave Length,  $\bar{x}_\lambda$ ,  $\bar{y}_\lambda$ ,  $\bar{z}_\lambda$ .*—The functions of wave length which serve to define the X, Y, and Z scales.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1929 to 1930, being revised in 1930. It was adopted as standard in 1930, published as standard from 1930 to 1937, but revised and republished as tentative from 1937 to 1939, being revised in 1939. It was again adopted as standard in 1939, published as standard from 1939 to 1942, but revised and republished as tentative in 1942.

<sup>3</sup> *Proceedings, Eighth Session, Commission Internationale de l'Eclairage, Cambridge, England, September, 1931, pp. 19-29.*

NOTE.—The function,  $\bar{y}_\lambda$ , is known as spectral luminosity. It evaluates the light-producing capacity, or luminous efficiency, of radiant energy.

(c) *Spectral Irradiance*,  $E_\lambda$ .—The density of radiant flux incident on an element of surface per unit wave length.

NOTE.—A source characterized by values of spectral irradiance constant throughout the spectrum is known as an "equal-energy source."

(d) *Tristimulus Designations*,  $X_o$ ,  $Y_o$ ,  $Z_o$ .—The color designations of the standard used in the spectrophotometric measurements of the test specimen.

(e) *Trichromatic Coefficients*,  $x$ ,  $y$ ,  $z$ .—Tristimulus designations expressed as fractions of their total, as follows:

$$x = \frac{X}{X + Y + Z}$$

$$y = \frac{Y}{X + Y + Z}$$

$$z = \frac{Z}{X + Y + Z}$$

(f) *Spectral Transmission*,  $T_\lambda$ .—The ratio of transmitted energy to the spectrally homogeneous energy incident on any test specimen.

(g) *Luminous Transmission*,  $T_s$ .—The ratio of transmitted to incident light.

NOTE.—Luminous transmission is a function of the spectral distribution of the incident energy. It may be calculated from spectral transmission,  $T_\lambda$ , and the spectral luminosity,  $\bar{y}_\lambda$ , as follows:

$$T_s = \frac{\sum_0^\infty T_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_0^\infty E_\lambda \bar{y}_\lambda \Delta\lambda}$$

(h) *Spectral Reflectance*.—The ratio of the reflected energy to the spectrally homogeneous energy incident on any test specimen.

NOTE.—Spectral reflectance depends upon the angular distribution of the incident energy.

(i) *Spectral Reflectivity*.—The spectral reflectance of a test specimen consisting of a layer of material of sufficient thickness that any increase would fail to change this ratio.

NOTE.—Spectral reflectivity depends upon the angular distribution of the incident energy.

(j) *Spectral Apparent Reflectance*.—The spectral reflectance which an ideal diffusing surface would need to possess in order to yield the same appearance as the test specimen under the same illuminating and viewing conditions.

NOTE.—Spectral apparent reflectance depends upon the angular distribution of the incident light and upon the direction of viewing.

(k) *Spectral Apparent Reflectivity*,  $R_\lambda$ .—The spectral reflectance which an ideal diffusing surface would need to possess in order to yield the same appearance as the test material under the same illuminating and viewing conditions.

NOTE.—Spectral apparent reflectivity depends upon the angular distribution of the incident light and upon the direction of viewing. It differs from spectral apparent reflectance only because it characterizes a material rather than a specimen. The ending "ivity" is customarily reserved for this meaning.

(l) *Luminous Reflectance*.—The ratio of the light reflected by any test specimen to that incident on it.

NOTE.—Luminous reflectance depends upon the spectral and angular distribution of the incident energy.

(m) *Luminous Reflectivity*.—The luminous reflectance of a test specimen consisting of a layer of material of sufficient thickness that any increase would fail to change this ratio.

NOTE.—Luminous reflectivity depends upon the spectral and angular distribution of the incident energy.

(n) *Luminous Apparent Reflectance*.—The luminous reflectance which a perfectly diffusing surface would need to possess in order to appear exactly as bright as the test specimen under the

same illuminating and viewing conditions.

NOTE.—Luminous apparent reflectance depends upon the spectral and angular distribution of the incident energy and upon the direction of viewing.

(o) *Luminous Apparent Reflectivity,  $R_s$ .*—The luminous reflectance which a perfectly diffusing surface would need to possess in order to appear exactly as bright as the test material under the same illuminating and viewing conditions.

NOTE.—Luminous apparent reflectivity depends upon the spectral and angular distribution of the incident energy and upon the direction of viewing. It may be calculated from spectral apparent reflectivity,  $R_\lambda$ , and the spectral luminosity,  $\bar{y}_\lambda$ , as follows:

$$R_s = \frac{\sum_0^\infty R_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda}{\sum_0^\infty E_\lambda \bar{y}_\lambda \Delta\lambda}$$

(p) *Spectral Characteristics.*—The appropriate quantity as a function of wave length by means of which the test specimen is characterized. Spectral transmission as a function of wave length characterizes a light-transmitting specimen; spectral apparent reflectivity characterizes a light-reflecting material; and spectral apparent reflectance characterizes a light-reflecting object.

### Apparatus

3. The apparatus shall consist of a spectrophotometer by means of which the spectral characteristics of the test specimen may, within the limits of the visible spectrum, be determined. Such apparatus may be of either the visual or the photoelectric type, but shall be suitable for determinations throughout the visible spectrum. The apparatus shall be so designed as to provide for the elimination of stray energy. This may be done by such means as the introduction of stray-energy filters or the use of two dispersing prisms.

### Standards

4. (a) For transparent specimens, the incident light shall be used as a standard relative to which the transmitted light is evaluated.

(b) For reflecting specimens, either of the following shall be used as a standard:

(1) The ideal, completely reflecting, completely diffusing surface, or

(2) A layer (at least 0.5 mm. in thickness) of magnesium oxide, freshly prepared by collecting the smoke from burning magnesium ribbon or shavings on a suitable surface.

NOTE.—For further information regarding the preparation of fumed standards, see "Preparation and Colorimetric Properties of a Magnesium-Oxide Reflectance Standard," *Letter Circular LC-547*, Nat. Bureau Standards, March, 1939.

### Illumination and Viewing

5. (a) The angular distribution of the light incident on the test specimen shall be specified, stating whether the illumination is diffuse, nearly diffuse, or nearly unidirectional. If nearly unidirectional, the direction and angular size of the source shall be specified. Unless there is a special reason for doing otherwise, light-transmitting specimens shall be illuminated only from directions near the perpendicular to the surface.

(b) If a test specimen be viewed by reflected light the angular distribution of the light incident on it shall be identical with that incident on any standard, such as a layer of magnesium oxide, with which it is compared.

(c) The direction and angular size of the viewing element shall be specified.

NOTE.—This method of measuring the spectral characteristics of a test specimen serves to specify those of its optical properties which are responsible for its color, subject, however, to the limitation that the color may depend upon the manner of illumination and viewing. For glossy samples in particular, it is important to specify the manner of illumination and viewing, and the most essential single characteristic is whether or not the specular component of reflected energy is included in the measurement.

Since glossy samples are customarily examined for color in such a way as to avoid specularly reflected light as much as possible, it is recommended that in these measurements also, the specular component be excluded by proper choice of illuminating and viewing conditions (such as 45-deg. illumination with perpendicular viewing, as recommended in 1931 by the International Commission on Illumination) or by application of a suitable correction. It should be noted that a light-reflecting specimen requires for specification of its appearance, in addition to the above, some measure of its gloss. This procedure is beyond the intended scope of this method and such measurement of gloss shall be made in accordance with the Tentative Method of Test for Specular Gloss of Paint Finishes (A.S.T.M. Designation: D 523) of the American Society for Testing Materials.<sup>4</sup>

### Calibrations

6. (a) The calibration of the photometer scale, or other intensity-measuring unit, shall be carefully checked at reasonable time intervals in a manner to insure accuracy over the entire range.

(b) The accuracy of the wave-length calibration shall be adequately checked at reasonable time intervals.

(c) The slit width shall be adjusted so that slit-width corrections are negligible; otherwise these corrections shall be applied.

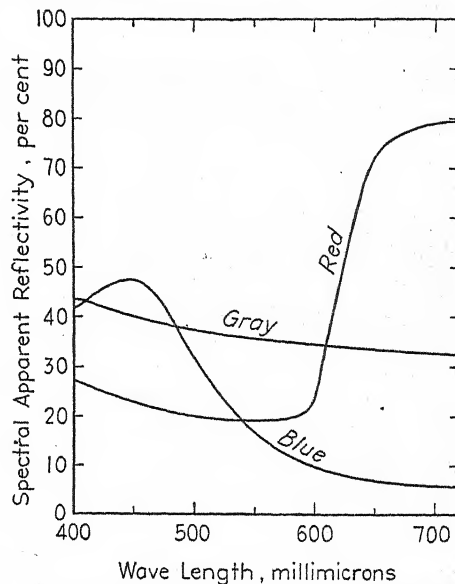
### Procedure

7. (a) The test specimen shall be handled carefully to avoid discoloration, and care shall be taken not to touch the area to be tested except for application of a suitable cleaning process. The condition of the test specimen before and after analysis shall be carefully noted and any change in the appearance of the surface of the tested area shall be recorded.

(b) Determination of spectral characteristics relative to the standard shall be made throughout such wave-length range within the visible spectrum and at such wave-length intervals that the curve drawn by interpolation and extra-

polarization would not be changed significantly by taking further readings.

NOTE.—Readings at 20  $m\mu$  intervals between 400 and 700  $m\mu$  are often sufficient; 10  $m\mu$  intervals between 380 and 770  $m\mu$  usually are sufficient. Typical curves of flat paints are shown in Fig. 1.



NOTE.—The red, gray, and blue paints were prepared by extending cadmium red, ivory black, and Prussian blue, respectively, with zinc white.

FIG. 1.—Typical Curves of Spectral Apparent Reflectivity.

(c) If spectral apparent reflectivity of a material is being determined, the test specimen shall consist of a layer of the material so thick that further increase in thickness will not cause a change in appearance (see Note 2, Section 1).

### Rejection

9. If the test specimen undergoes a visible change during the analysis, the readings shall be rejected (see Section 7 (a)).

### Report

10. The report of the spectral characteristics of a test specimen shall consist of the following:

(a) The spectral transmission,  $T_\lambda$ , of a light-transmitting object, or the spec-

tral apparent reflectance of a light-reflecting object, or the spectral apparent reflectivity,  $R_\lambda$ , of a light-reflecting material.

NOTE.—If the test specimen is a translucent object which both transmits and reflects appreciable quantities of light, it may be necessary to report both its spectral transmission and its spectral apparent reflectance.

(b) The manner of illumination and manner of viewing, as specified in Section 5 (a) and (c).

### General Plan of Color Designation

11. The color of a nonself-luminous specimen depends both upon the spectral composition of the illuminant and upon the spectral characteristics of the specimen itself. The method of designating the color of a material or object under a particular illuminant is to evaluate the light leaving it on each of three scales, an  $X$ -scale, a  $Y$ -scale, and a  $Z$ -scale. The color of a light-transmitting object for a particular illuminant should be designated by its luminous transmission,  $T_\lambda$ , and trichromatic coefficients,  $x$  and  $y$ , for that illuminant. Similarly, the color of a reflecting material under a particular illuminant should be designated by its luminous apparent reflectivity,  $R_\lambda$ , and its trichromatic coefficients,  $x$  and  $y$ , for that illuminant. Also, the color of a reflecting object under a particular illuminant should be designated by its luminous apparent reflectance and its trichromatic coefficients,  $x$  and  $y$ , for that illuminant. These quantities may be found from the values of  $X$ ,  $Y$ , and  $Z$  for the test specimen and standard as described in Sections 12 and 13.

### Standard Illuminants

12. Standard illuminant A shall be used if the color of the test specimen in incandescent lamplight is of interest, standard illuminant B for the color of the specimen in sunlight, and standard

illuminant C for the color of the specimen in average daylight.<sup>5</sup> The relative spectral irradiance,  $E_\lambda$ , of these three

TABLE I.—RELATIVE SPECTRAL IRRADIANCE OF THE THREE STANDARD ILLUMINANTS, A, B, AND C.

Wave Length, $m\mu$	$E_A$	$E_B$	$E_C$	Wave Length, $m\mu$	$E_A$	$E_B$	$E_C$
380	9.79	22.40	33.00	580	114.44	101.00	97.80
385	10.90	26.85	39.92	585	118.08	100.07	95.43
390	12.09	31.30	47.40	590	121.73	99.20	93.20
395	13.36	36.18	55.17	595	125.39	98.44	91.22
400	14.71	41.30	63.30	600	129.04	98.00	89.70
405	16.15	46.62	71.81	605	132.70	98.08	88.83
410	17.68	52.10	80.60	610	136.34	98.50	88.40
415	19.29	57.70	89.53	615	139.99	99.06	88.19
420	21.00	63.20	98.10	620	143.62	99.70	88.10
425	22.79	68.37	105.80	625	147.23	100.36	88.06
430	24.67	73.10	112.40	630	150.83	101.00	88.00
435	26.64	77.31	117.75	635	154.42	101.56	87.86
440	28.70	80.80	121.50	640	157.98	102.20	87.80
445	30.85	83.44	123.45	645	161.51	103.05	87.99
450	33.09	85.40	124.00	650	165.03	103.90	88.20
455	35.41	86.88	123.60	655	168.51	104.59	88.20
460	37.82	88.30	123.10	660	171.96	105.00	87.90
465	40.30	90.08	123.30	665	175.38	105.08	87.22
470	42.87	92.00	123.80	670	178.77	104.90	86.30
475	45.52	93.75	124.09	675	182.12	104.55	85.30
480	48.25	95.20	123.90	680	185.43	103.90	84.00
485	51.04	96.23	122.92	685	188.70	102.84	82.21
490	53.91	96.50	120.70	690	191.93	101.60	80.20
495	56.85	95.71	116.90	695	195.12	100.38	78.24
500	59.86	94.20	112.10	700	198.26	99.10	76.30
505	62.93	92.37	106.98	705	201.36	97.70	74.36
510	66.06	90.70	102.30	710	204.41	96.20	72.40
515	69.25	89.65	98.81	715	207.41	94.60	70.40
520	72.50	89.50	96.90	720	210.36	92.90	68.30
525	75.79	90.43	96.78	725	213.26	91.10	66.30
530	79.13	92.20	98.00	730	216.12	89.40	64.40
535	82.52	94.46	99.94	735	218.92	88.00	62.83
540	85.95	96.90	102.10	740	221.66	86.90	61.50
545	89.41	99.16	103.95	745	224.36	85.90	60.20
550	92.91	101.00	105.20	750	227.00	85.20	59.20
555	96.44	102.20	105.67	755	229.58	84.80	58.50
560	100.00	102.80	105.30	760	232.11	84.70	58.10
565	103.58	102.92	104.11	765	234.59	84.90	58.00
570	107.18	102.60	102.30	770	237.01	85.40	58.20
575	110.80	101.90	100.15	775	239.37	86.10	58.50
580	114.44	101.00	97.80	780	241.67	87.00	59.10

standard illuminants throughout the visible spectrum is given in Table I, and the trichromatic coefficients,  $x$ ,  $y$ ,  $z$ , of

<sup>5</sup> For information regarding the procurement of lamps and liquid filters required to duplicate these standard illuminants, reference should be made to the following articles: *Proceedings, Eighth Session, Commission Internationale de l'Eclairage*, Cambridge, England, September, 1931, pp. 19-29.

A. C. Hardy, "Handbook of Colorimetry," p. 16, Technology Press, Cambridge, Mass. (1936).

D. B. Judd, "The 1931 I.C.I. Standard Observer and Coordinate System for Colorimetry," *Journal, Optical Soc. America*, Vol. 23, p. 359 (1933).

R. Davis and K. S. Gibson, "Filters for the Reproduction of Sunlight and Daylight and the Determination of Color Temperature," *Miscellaneous Publication No. 114*, Nat. Bureau Standards (1931).

the standard illuminants are given in Table II.

TABLE II.—TRICHROMATIC COEFFICIENTS OF THE STANDARD ILLUMINANTS.

Standard Illuminant	Trichromatic Coefficients		
	$x$	$y$	$z$
A.....	0.4476	0.4075	0.1449
B.....	0.3484	0.3516	0.3000
C.....	0.3101	0.3163	0.3736

## Procedure

13. (a) The spectral composition of the light leaving a nonself-luminous reflecting material is found by multiplying, for each wave length of the visible spectrum, the spectral irradiance,  $E_\lambda$ , of the illuminant by the spectral apparent reflectivity,  $R_\lambda$ , of the material; that is, the product,  $R_\lambda E_\lambda$ , is found throughout the visible spectrum (380 to 770  $m\mu$ ). If magnesium oxide is used as the standard, the spectral apparent reflectivity of the test specimen should be expressed relative to that for magnesium oxide.

NOTE 1.—The evaluation of  $X$ ,  $Y$ , and  $Z$  for a transparent object viewed by transmitted light, or for an opaque object viewed by reflected light, is found in a way strictly analogous to that for an opaque layer of material viewed by reflected light. The spectral transmission,  $T_\lambda$ , and spectral apparent reflectance of an object serve the same purpose as the spectral apparent reflectivity,  $R_\lambda$ , of a reflecting material.

(b) The light leaving the specimen of spectral composition,  $R_\lambda E_\lambda$ , is evaluated on the  $X$ -scale for each part of the spectrum by multiplying the corresponding  $R_\lambda E_\lambda$  by the  $X$ -value for unit irradiance for that part of the spectrum. This light is also evaluated on the  $Y$ , and  $Z$  scales in the same way. Table III gives the  $X$ ,  $Y$ , and  $Z$  values for unit irradiance throughout the visible spectrum for each 5- $m\mu$  interval. These values are identified by the symbols,  $\bar{x}_\lambda$ ,  $\bar{y}_\lambda$ ,  $\bar{z}_\lambda$ , respectively. From this

step the products,  $R_\lambda E_\lambda \bar{x}_\lambda$ ,  $R_\lambda E_\lambda \bar{y}_\lambda$ , and  $R_\lambda E_\lambda \bar{z}_\lambda$  are found throughout the visible spectrum.

(c) The light leaving the specimen of spectral composition,  $R_\lambda E_\lambda$ , is evaluated on the  $X$ -scale for the whole spectrum by adding together the values of  $X$  found for the intervals of the spectrum separately. The light is also evaluated on the  $Y$  and  $Z$  scales in the same way. From this step are found the three numbers,  $X$ ,  $Y$ , and  $Z$ :

$$X = \sum_0^\infty R_\lambda E_\lambda \bar{x}_\lambda \Delta\lambda$$

$$Y = \sum_0^\infty R_\lambda E_\lambda \bar{y}_\lambda \Delta\lambda$$

$$Z = \sum_0^\infty R_\lambda E_\lambda \bar{z}_\lambda \Delta\lambda$$

NOTE 2.—For materials of usual spectral selectivity, an accurate evaluation of  $X$ ,  $Y$ , and  $Z$  is obtainable by setting  $\Delta\lambda = 10 m\mu$ ; but for some materials it is found necessary to take  $\Delta\lambda = 5 m\mu$  or even less. For interpolated values of  $\bar{x}_\lambda$ ,  $\bar{y}_\lambda$ , and  $\bar{z}_\lambda$  for each millimicron from 380 to 770  $m\mu$ , see A. C. Hardy, "Handbook of Colorimetry," Technology Press, Cambridge (1936). The selected-ordinate method of calculating  $X$ ,  $Y$ , and  $Z$ , in many circumstances the most rapid, is also described there.<sup>6,7</sup>

(d) Calculate the luminous apparent reflectivity,  $R_s$ , and two of the three

<sup>6</sup> For further information regarding methods of carrying out these calculations, reference should be made to the following articles:

T. Smith, "Condensed Tables for Colour Computation," *Proceedings*, Physical Soc., London, Vol. 46, p. 372 (1934).

D. Nickerson, "Disk Colorimetry; Including a Comparison of Methods for Computing Tristimulus Values for Certain Disks," *Journal*, Optical Soc. America, Vol. 25, p. 253 (1935).

H. W. Swanik and M. G. Mellon, "A Calculator for Obtaining Tristimulus Values from Spectrophotometric Data," *Journal*, Optical Soc. America, Vol. 27, p. 414 (1937).

F. W. Sears, "An Improved Calculator for Obtaining Tristimulus Values from Spectrophotometric Curves," *Journal*, Optical Soc. America, Vol. 29, p. 77 (1939).

J. A. Van den Akker, "A Mechanical Integrator for Evaluating the Integral of the Product of Two Functions and Its Application to the Computation of I.C.I. Color Specifications from Spectrophotometric Curves," *Journal*, Optical Soc. America, Vol. 29, p. 364 (1939).

<sup>7</sup> For attachments to the spectrophotometer to perform these calculations automatically, reference should be made to the following articles:

A. C. Hardy, "A New Recording Spectrophotometer," *Journal*, Optical Soc. America, Vol. 25, p. 305 (1935).

J. Razek, "Electrical Device for the Determination of Trilinear Coordinates for the Razek-Mulder Color Analyzer," *Journal*, Optical Soc. America, Vol. 24, p. 54 (1934).

trichromatic coefficients  $x$  and  $y$ , as standard (see Table II) are found by calculating the following ratios:

$$R_s = \frac{Y}{Y_s}$$

$$x \equiv \frac{X}{X + Y + Z}$$

$$y \equiv \frac{Y}{X + Y + Z}$$

$$\frac{X_s}{X_s + Y_s + Z_s}, \text{ and}$$

$$\frac{Y_s}{X_s + Y_s + Z_s}$$

Table III gives the trichromatic coeffi-

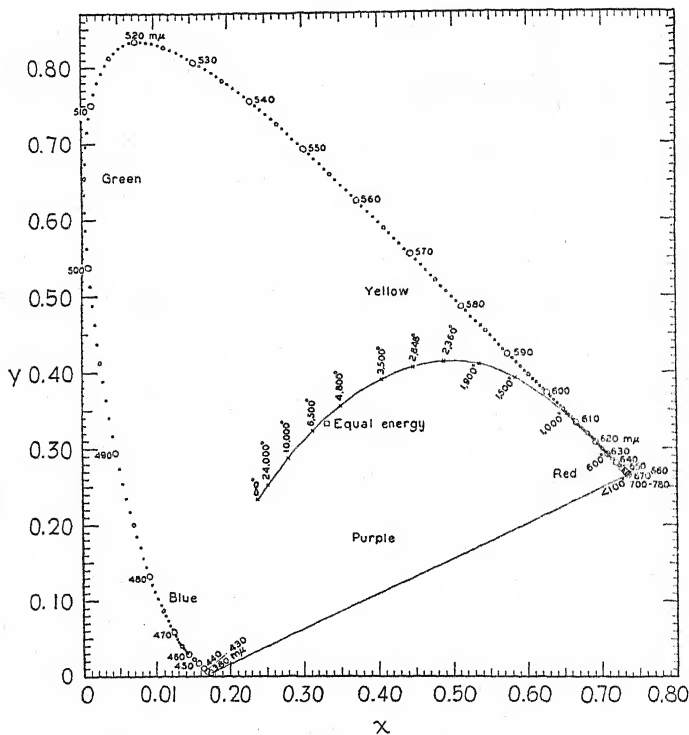


FIG. 2.—The  $(x, y)$ -Diagram Showing the Spectrum Colors.

It is customary to use a plot of  $y$  against  $x$  in rectangular coordinates (called a chromaticity diagram or mixture diagram) as an aid in determining the relation of the color of the specimen to known colors. For this purpose the colors of the spectrum and that of the standard are most frequently used. The trichromatic coefficients of the

coefficients,  $x$ ,  $y$ ,  $z$ , of the spectrum colors, and the spectrum colors on the  $(x, y)$ -diagram are shown in Fig. 2.

NOTE 3.—If the specimen is a transparent object, the ratio,  $Y/Y_s$ , should be designated the luminous transmission; if it is a reflecting object consisting of a top translucent layer backed by a different material, the ratio  $Y/Y_s$  should be designated the luminous apparent reflectance.



## Report

14. The report of the color designation of the test specimen shall consist of the following:

(a) The trichromatic coefficients,  $x$  and  $y$ .

specimen is a reflecting material, a reflecting object, or a light-transmitting object, respectively.

(c) The manner of illumination and manner of viewing as specified in Section 5 (a) and (c).

TABLE III.—THE 1931 I.C.I. STANDARD OBSERVER.

Trichromatic Coefficients			Wave Length, $m\mu$	Tristimulus Specifications of the Equal-energy Spectrum			Trichromatic Coefficients			Wave Length, $m\mu$	Tristimulus Specifications of the Equal-energy Spectrum		
$x$	$y$	$z$		$\bar{x}_\lambda$	$\bar{y}_\lambda$	$\bar{z}_\lambda$	$x$	$y$	$z$		$\bar{x}_\lambda$	$\bar{y}_\lambda$	$\bar{z}_\lambda$
0.1741	0.0050	0.8209	380	0.0014	0.0000	0.0065	0.5125	0.4866	0.0009	580	0.9163	0.8700	0.0017
0.1740	0.0050	0.8210	385	0.0022	0.0001	0.0105	0.5448	0.4544	0.0008	585	0.9786	0.8163	0.0014
0.1738	0.0049	0.8213	390	0.0042	0.0001	0.0201	0.5752	0.4242	0.0006	590	1.0263	0.7570	0.0011
0.1736	0.0049	0.8215	395	0.0076	0.0002	0.0362	0.6029	0.3965	0.0006	595	1.0567	0.6949	0.0010
0.1733	0.0048	0.8219	400	0.0143	0.0004	0.0679	0.6270	0.3725	0.0005	600	1.0622	0.6310	0.0008
0.1730	0.0048	0.8222	405	0.0232	0.0006	0.1102	0.6482	0.3514	0.0004	605	1.0456	0.5668	0.0006
0.1726	0.0048	0.8226	410	0.0435	0.0012	0.2074	0.6658	0.3340	0.0002	610	1.0026	0.5030	0.0003
0.1721	0.0048	0.8231	415	0.0776	0.0022	0.3713	0.6801	0.3197	0.0002	615	0.9384	0.4412	0.0002
0.1714	0.0051	0.8235	420	0.1344	0.0040	0.6456	0.6915	0.3083	0.0002	620	0.8544	0.3810	0.0002
0.1703	0.0058	0.8239	425	0.2148	0.0073	1.0391	0.7006	0.2993	0.0001	625	0.7514	0.3210	0.0001
0.1689	0.0069	0.8242	430	0.2839	0.0116	1.3856	0.7079	0.2920	0.0001	630	0.6424	0.2650	0.0000
0.1669	0.0086	0.8245	435	0.3285	0.0168	1.6230	0.7140	0.2859	0.0001	635	0.5419	0.2170	0.0000
0.1644	0.0109	0.8247	440	0.3483	0.0230	1.7471	0.7190	0.2809	0.0001	640	0.4479	0.1750	0.0000
0.1611	0.0138	0.8251	445	0.3481	0.0298	1.7826	0.7230	0.2770	0.0000	645	0.3608	0.1382	0.0000
0.1566	0.0177	0.8257	450	0.3362	0.0380	1.7721	0.7260	0.2740	0.0000	650	0.2835	0.1070	0.0000
0.1510	0.0227	0.8263	455	0.3187	0.0480	1.7441	0.7283	0.2717	0.0000	655	0.2187	0.0816	0.0000
0.1440	0.0297	0.8263	460	0.2908	0.0600	1.6692	0.7300	0.2700	0.0000	660	0.1649	0.0610	0.0000
0.1355	0.0399	0.8246	465	0.2511	0.0739	1.5281	0.7311	0.2689	0.0000	665	0.1212	0.0446	0.0000
0.1241	0.0578	0.8181	470	0.1954	0.0910	1.2876	0.7320	0.2680	0.0000	670	0.0874	0.0320	0.0000
0.1096	0.0868	0.8036	475	0.1421	0.1126	1.0419	0.7327	0.2673	0.0000	675	0.0636	0.0232	0.0000
0.0913	0.1327	0.7760	480	0.0956	0.1390	0.8130	0.7334	0.2666	0.0000	680	0.0468	0.0170	0.0000
0.0687	0.2007	0.7306	485	0.0580	0.1693	0.6162	0.7340	0.2660	0.0000	685	0.0329	0.0119	0.0000
0.0454	0.2950	0.6596	490	0.0320	0.2080	0.4652	0.7344	0.2656	0.0000	690	0.0227	0.0082	0.0000
0.0235	0.4127	0.5638	495	0.0147	0.2586	0.3533	0.7346	0.2654	0.0000	695	0.0158	0.0057	0.0000
0.0082	0.5384	0.4534	500	0.0049	0.3230	0.2720	0.7347	0.2653	0.0000	700	0.0114	0.0041	0.0000
0.0039	0.6548	0.3413	505	0.0024	0.4073	0.2123	0.7347	0.2653	0.0000	705	0.0081	0.0029	0.0000
0.0139	0.7502	0.2359	510	0.0093	0.5030	0.1582	0.7347	0.2653	0.0000	710	0.0058	0.0021	0.0000
0.0389	0.8120	0.1491	515	0.0291	0.6082	0.1117	0.7347	0.2653	0.0000	715	0.0041	0.0015	0.0000
0.0743	0.8338	0.0919	520	0.0633	0.7100	0.0782	0.7347	0.2653	0.0000	720	0.0029	0.0010	0.0000
0.1142	0.8262	0.0596	525	0.1096	0.7932	0.0573	0.7347	0.2653	0.0000	725	0.0020	0.0007	0.0000
0.1547	0.8059	0.0394	530	0.1655	0.8620	0.0422	0.7347	0.2653	0.0000	730	0.0014	0.0005	0.0000
0.1929	0.7816	0.0255	535	0.2257	0.9149	0.0298	0.7347	0.2653	0.0000	735	0.0010	0.0004	0.0000
0.2296	0.7543	0.0161	540	0.2904	0.9540	0.0203	0.7347	0.2653	0.0000	740	0.0007	0.0003	0.0000
0.2658	0.7243	0.0099	545	0.3597	0.9803	0.0134	0.7347	0.2653	0.0000	745	0.0005	0.0002	0.0000
0.3016	0.6923	0.0061	550	0.4334	0.9950	0.0087	0.7347	0.2653	0.0000	750	0.0003	0.0001	0.0000
0.3373	0.6589	0.0038	555	0.5121	1.0002	0.0057	0.7347	0.2653	0.0000	755	0.0002	0.0001	0.0000
0.3731	0.6245	0.0024	560	0.5945	0.9950	0.0039	0.7347	0.2653	0.0000	760	0.0002	0.0001	0.0000
0.4087	0.5896	0.0017	565	0.6784	0.9786	0.0027	0.7347	0.2653	0.0000	765	0.0001	0.0000	0.0000
0.4441	0.5547	0.0012	570	0.7621	0.9520	0.0021	0.7347	0.2653	0.0000	770	0.0001	0.0000	0.0000
0.4788	0.5202	0.0010	575	0.8425	0.9154	0.0018	0.7347	0.2653	0.0000	775	0.0000	0.0000	0.0000
0.5125	0.4866	0.0009	580	0.9163	0.8700	0.0017	0.7347	0.2653	0.0000	780	0.0000	0.0000	0.0000
Totals .....											21.3713	21.3714	21.3715

(b) The luminous apparent reflectivity,  $R_s$ , the luminous apparent reflectance, or the luminous transmission,  $T_s$ , depending upon whether the test

(d) The illuminant to which the color designation of the test specimen applies; that is, whether the standard illuminant, A, B, or C, or some other illuminant.



# *Standard Method of Test for*

## SURFACE IRREGULARITIES OF FLAT TRANSPARENT PLASTIC SHEET<sup>1</sup>



A.S.T.M. Designation: D 637-43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 637; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method of test is intended for the measurement of the surface irregularities of flat transparent plastic sheets that are ordinarily used to cover openings through which visual and instrumental observations are made.

(b) This method measures the distortion and the deviation of line of sight through flat sheets of transparent plastics. The method makes use of the prismatic or optical wedge deflection of a beam of light as it passes through a distortion spot or wave in the body of or on the surface of the material being inspected.

### Description of Terms

2. (a) *Displacement Factor*.—Displacement factor is the maximum movement (in inches) of the image of the cross, divided by the distance (in feet) from the projector to the screen, multiplied by 1000.

(b) *Frequency of Image Movement*.—Frequency of image movement shall be described as (1) irregular or wavy, (2) frequent, or (3) single shift.

(c) *Pattern Distance*.—Pattern distance is the maximum distance (in integer multiples of 5 in.) from the screen at which the specimen can be held without producing a sharply defined pattern of its minor surface irregularities.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Projector*.—A good quality lantern slide projector or similar assembly of light source and lenses that is capable of producing a sharply defined image on a screen at a distance of 25 ft. The objective lens of this system shall have an aperture approximately 2-in. in diameter and a focal length of approximately 12 in.

(b) *Slide*.—A transparent slide on which have been ruled two very fine black lines crossing at right angles at the center of the slide. This slide may be prepared by photographing a drawing

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1943.

or by directly ruling fine lines on some suitable transparent medium.

(c) *Screen*.—A square white screen measuring about 5 ft. on a side. This screen shall have a symmetrical cross ruled on it consisting of seven horizontal parallel fine lines spaced 0.5 in. apart and seven vertical parallel fine lines spaced 0.5 in. apart. These lines shall be as fine as it is possible to make them and still have them distinctly visible at a distance of 25 ft. A width of about  $\frac{1}{16}$  in. is suggested.

NOTE.—An alternative form of target may be preferred, consisting of concentric circles having radii increasing by 1 in. from a 1-in. radius for the innermost circle, ruled in the center of a piece of white cardboard 1 ft. square. It is suggested that the first circle be ruled with a line thickness of  $\frac{1}{8}$  in., the second with a line thickness of  $\frac{1}{16}$  in., the third with a line thickness of  $\frac{1}{8}$  in., and so on.

(d) *Supports*.—A suitable means of rigidly supporting the projector and the screen.

(e) *Dark Room*.—A slightly darkened or dark room of sufficient length to accommodate the test setup. It has been found that an illumination level of not over 10 foot-candles in the room will be satisfactorily dark when an ordinary lantern slide projector is being used.

#### Assembly of Apparatus

4. (a) The screen shall be placed 25 ft. from the front lens of the projector and perpendicular to the direction of projection.

(b) The cross-ruled slide shall be placed in the projector and the projector and its lens system adjusted to throw a sharply defined image of the cross directly on top of the center cross ruled on the screen.

NOTE.—If the alternate form of target is used, the projector shall be adjusted so that the center of the image of the cross falls on the center of the circles.

#### Test Specimen

5. The test specimen shall consist of any flat sheet of a transparent plastic.

#### Procedure

6. (a) The test specimen shall be held with its plane perpendicular to the direction of projection and in front of the projector at a distance of approximately 12 in. from the front lens and shall be moved so that the entire area inside of a 1-in. border around the sheet is surveyed by the beam of light from the projector system. While the specimen is being moved about, the screen shall be observed for movement of the projected image of the cross. The maximum amount and nature or frequency of movement of the image shall be noted.

(b) After the specimen has been completely surveyed in the position near the projector, it shall be moved parallel to and at a distance of 5 in. from the screen. The specimen shall then be moved back and forth parallel to the screen and any projected images of minor irregularities that are visible on the screen shall be observed. The specimen shall then be moved to positions 10 in., 15 in., or 20 in. (integer multiples of 5 in.) from the screen until a pattern is observed. The maximum distance from the screen at which the specimen can be held without producing a pattern of its minor surface irregularities shall be noted.

#### Report

7. The report shall include the following:

- (1) The displacement factor,
- (2) The frequency and nature of the shift of the image, and
- (3) The pattern distance in an integer multiple of 5 in.

*Standard Method of Test for*  
COEFFICIENT OF LINEAR THERMAL EXPANSION  
OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 696 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 696; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Scope<sup>2a</sup>**

1. The thermal expansion of a plastic is composed of a reversible component on which are superimposed changes in length due to changes in moisture content, curing (degree of polymerization), loss of plasticizer or solvents, release of stresses, and other factors. This method of test is intended for determining the reversible linear thermal expansion under exclusion of these accidental factors. In general, it will not be possible to exclude all these spurious factors and for this reason the method can be expected to give only approximate values. The dilatometer described in the Appendix to this method has the advantage of simplicity, but it is not intended to be used when results of great accuracy are desired; it is also not applicable to very soft plastics such as elastomers.

**Apparatus**

2. The apparatus shall consist of the following:

(a) *Dilatometer*.—Any generally accepted apparatus that will measure the coefficient of linear thermal expansion with an accuracy of plus or minus 10 per cent may be used. A simple fused-quartz-tube dilatometer similar in principle to the apparatus indicated in the Standard Method of Test for Linear Expansion of Metals (A.S.T.M. Designation: B 95) of the American Society for Testing Materials<sup>3</sup> is described in the Appendix. The accuracy of the apparatus used shall be such that it measures the change in length over the temperature interval under consideration within plus or minus 2 per cent. The device that measures the expansion shall not exert a stress of more than 10 psi. on the specimen. It is the intent of this limitation that the thrust of the device in contact with the specimen, shall not distort the specimen or appreciably indent its surface.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

<sup>2a</sup> Editorially revised in 1946.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part I-B.

(b) *Scale or Caliper*.—A scale or caliper capable of measuring the test length of the specimen with an accuracy of plus or minus 0.5 per cent.

(c) *Temperature Bath*.—A liquid bath for controlling the temperature of the specimen (see Note, Section 5(e)). The bath shall be arranged in such a way that uniform temperature throughout the specimen is assured. Means shall be provided for stirring the bath and for controlling its temperature within plus or minus 0.2 C. at the temperatures used.

NOTE.—It is preferable to avoid contact between the bath liquid and the test specimen. If this is not practicable care must be taken to select a liquid that will not affect the physical properties of the material under test.

### Test Specimens

3. (a) The samples from which test specimens are to be cut or shaped shall be molded or cast by methods and under conditions that give a minimum of strains or molecular anisotropy. This requirement applies also if the specimens are molded to their finished shape. When testing materials which exhibit different properties in different directions, specimens shall be cut along the principal axes of anisotropy and the coefficient of linear thermal expansion shall be measured separately along the different axes.

(b) When using the dilatometer described in the Appendix, the test specimen may be round or square and should fit easily into the outer tube of the dilatometer without excessive play. The specimen may be 2 to 4 in. in length but its length will be governed to some extent by the sensitivity of the dial gage, the expansion expected, and the accuracy desired. The ends of the specimen shall be cut perpendicular to the axis of the specimen. Before the specimen is placed in the dilatometer, its ends shall be protected against indentation by means of thin steel plates cemented to them.

### Conditioning Specimens

4. Test specimens shall be conditioned in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials.<sup>4</sup>

### Procedure

5. (a) The nature of most plastics and the construction of the common type of dilatometer make  $-30$  C. to  $+30$  C. a convenient temperature range for linear thermal expansion measurements of plastics. This range covers the temperatures in which plastics are commonly used. Any extension or subdivision of this temperature range shall be made in steps of 30 C. When extending the range upward particular attention shall be paid to the factors mentioned in Section 1 and special investigations may be required to avoid excessive errors. The coefficient of linear thermal expansion of a few plastics is known to change rather abruptly at a certain temperature. To find this temperature it will be necessary to measure the coefficient of linear thermal expansion in narrow temperature limits, or to observe the rate of expansion during a steady rise in temperature of the specimen. Once such a transition point has been found, a separate coefficient of expansion for a temperature range below and above the transition point shall be determined.

(b) The length of the conditioned test specimen shall be measured at room temperature.

(c) The conditioned specimen shall be mounted in the dilatometer. The temperature of the bath shall be maintained at  $-30 \pm 0.2$  C. until the temperature of the specimen reaches the temperature of the bath. The time required shall be determined by preliminary measure-

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

ments of the inside temperature of the specimen. The reading of the dial indicator or other device that indicates the expansion of the specimen shall be recorded.

(d) With the temperature at  $+30 \pm 0.2$  C., the procedure described in Paragraph (c) shall be repeated.

(e) With the temperature again at  $-30 \pm 0.2$  C., the procedure described in Paragraph (c) shall be repeated.

**NOTE.**—When the construction of the apparatus permits, it may be more convenient to use alternately two baths at the proper temperatures. Care should be taken not to disturb the apparatus during the transfer from one bath to the other. The use of two baths is preferred because it will reduce the time required to bring the specimen to the desired temperature. The test in general should be conducted in as short a time as possible to avoid changes in physical properties during long exposures to high and low temperatures.

(f) If the change in length of the specimen due to heating does not agree with the change in length due to cooling within 10 per cent of their average, the cause of the discrepancy shall be investigated and if possible eliminated.

#### Calculation and Report

6. (a) *Calculation.*—The average coefficient of linear thermal expansion shall be calculated as follows:

$$\alpha = \frac{\Delta L}{L \times T}$$

where:

$\alpha$  = coefficient of linear thermal expansion per degree Centigrade,

$\Delta L$  = average of changes in length of test specimen due to heating and cooling,

$L$  = length of test specimen at room temperature ( $\Delta L$  and  $L$  being measured in the same units), and

$T$  = temperature difference in degrees Centigrade over which the changes in length of the specimen are measured.

(b) *Report.*—The report shall include the following:

(1) Designation of material, including name of manufacturer and information on composition when known,

(2) Method of preparation of test specimen,

(3) Form and dimensions of test specimen,

(4) Identification of the type of apparatus used,

(5) Temperatures between which the coefficient of linear thermal expansion has been determined,

(6) Average coefficient of linear thermal expansion per degree Centigrade, and

(7) Complete description of any unusual behavior of the specimen, for example, differences of more than 10 per cent in measured values of expansion and contraction.

(See Appendix, p. 233)

## APPENDIX

QUARTZ-TUBE DILATOMETER<sup>5</sup>

The quartz-tube dilatometer shown in Fig. 1 consists of the following:

**Outer Tube.**—An outer tube of fused quartz 20 in. in length  $\frac{1}{2}$  in. in inside diameter, and about 2 mm. in wall thickness. The tube is sealed on the lower end, the sealing surface being convex on the inside to provide one-point contact with the thin steel plate cemented to the lower end of the specimen.

**Inner Tube.**—An inner tube of fused quartz 16 to 18 in. in length with an outside diameter that allows this tube to fit snugly inside the outer tube without binding. The length of the inner tube is determined by the length of the specimen, since the upper ends of the inner and outer tubes should be at the same level when the specimen is in place. One end of the inner tube is closed with a hemispherical seal to provide one-point contact with the thin steel plate cemented to the upper end of the specimen. To the end of the inner tube is attached a thin steel plate or is fused a quartz plate which serves as a rest for the foot of the dial gage. The wall thickness of the inside quartz tube should be as small as practical in order to reduce the load on the specimen.

**Dial Gage.**—A dial gage mounted on a bracket which is securely fastened to the upper end of the outer tube. The dial gage is preferably calibrated to 0.0001 in. per division. The stem of the dial gage should be concentric with the quartz tube.

**Mounting.**—The dilatometer is mounted vertically on a solid support in such a way that the temperature bath can readily be changed without disturbing the dilatometer.

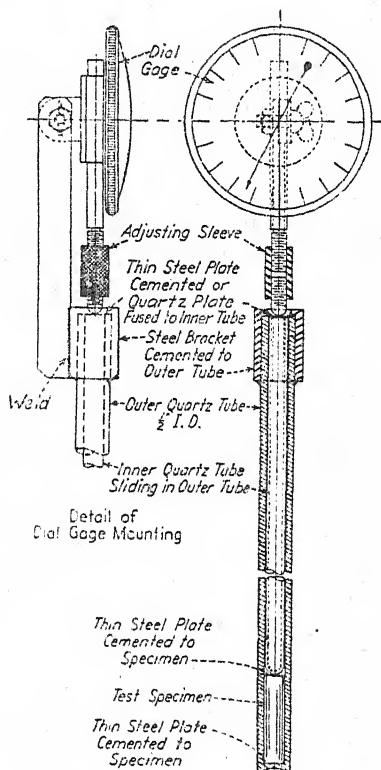


FIG. 1.—Quartz-Tube Dilatometer.

<sup>5</sup> Editorially revised, April, 1945.

# Standard Method of Test for WATER ABSORPTION OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 570 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 570; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method of test covers the procedure for determining the relative rate of absorption of water by plastics when immersed. The method is intended to apply to the testing of all types of plastics, including cast, hot-molded, and cold-molded resinous products, and both homogeneous and laminated plastics in rod and tube form, and in sheets 0.005 in. or greater in thickness.

NOTE 1.—The moisture content of a plastic is very intimately related to such properties as electrical insulation resistance, dielectric losses, mechanical strength, appearance, and dimensions. The effect upon these properties of change in moisture content due to water absorption depends largely on the type of exposure (by immersion in water or by exposure to high humidity), shape of the part, and inherent properties of the plastic. With nonhomogeneous materials, such as laminated forms, the rate of water absorption may be widely different through each edge and surface. Even for otherwise homogeneous materials, it may be slightly greater through cut edges than through molded surfaces. Consequently, attempts to correlate water absorption with surface area

must generally be limited to closely related materials and to similarly shaped specimens. For materials of widely varying density, relation between water absorption values on a volume as well as a weight basis may need to be considered.

NOTE 2.—The test for rate of water absorption has two chief functions: first, as a guide to the proportion of water absorbed by a material and consequently, in those cases where the relationships between moisture and electrical or mechanical properties, dimensions, or appearance have been determined, as a guide to the effects of exposure to water or humid conditions on such properties; and second, as a control test on the uniformity of a product. This second function is particularly applicable to sheet, rod, and tube forms when the test is made on the finished product.

## Apparatus

2. The apparatus shall consist of the following:

(a) *Balance*.—An accurate chemical balance.

(b) *Oven*.—An oven capable of maintaining uniform temperatures of  $50 \pm 3$  C. ( $122 \pm 5.4$  F.) and of 105 to 110 C. (221 to 230 F.).

## Test Specimens

3. (a) The test specimen for molded plastics shall be in the form of a disk

<sup>1</sup> Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

2 in. (5.08 cm.) in diameter and  $\frac{1}{8}$  in. (0.32 cm.) in thickness (Note). Permissible variations in thickness are plus or minus 0.007 in. for hot-molded and plus or minus 0.012 in. for cold-molded or cast materials.

NOTE.—The disk mold prescribed in Section 3 of the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (A.S.T.M. Designation: D 647) of the American Society for Testing Materials<sup>3</sup> is suitable for molding disk test specimens of thermosetting materials but not thermoplastic materials.

(b) The test specimen for sheets shall be in the form of a bar 3 in. (7.62 cm.) in length by 1 in. (2.54 cm.) in width by the thickness of the material. When comparison of absorption values with molded plastics is desired, specimens  $\frac{1}{8}$  in. in thickness should be used. Permissible variations in thickness of the  $\frac{1}{8}$ -in. thick specimen shall be plus or minus 0.008 in. except for asbestos-fabric-base phenolic laminated materials or other materials which have greater standard commercial tolerances.

(c) The test specimen for rods shall be 1 in. in length for rods 1 in. in diameter or under, and  $\frac{1}{2}$  in. in length for larger diameter rods. The diameter of the specimen shall be the diameter of the finished rod.

(d) The test specimen for tubes less than 3 in. in inside diameter shall be the full section of the tube and 1 in. in length. For tubes 3 in. or more in inside diameter, a rectangular specimen shall be cut 3 in. in length in the circumferential direction of the tube and 1 in. in width lengthwise of the tube.

(e) The test specimens for sheets, rods, and tubes shall be machined, sawed, or sheared from the sample so as to have smooth edges free from cracks. The cut edges shall be made smooth by finishing with No. 0 or finer sandpaper or

emery cloth. Sawing, machining, and sandpapering operations shall be slow enough so that the material is not heated appreciably (Note).

NOTE.—If there is any oil on the surface of the specimen when received or as a result of machining operations, wash the specimen with a cloth wet with gasoline to remove oil, wipe with a dry cloth, and allow to stand in air for 2 hr. to permit evaporation of the gasoline. If gasoline attacks the plastic, use some suitable solvent or detergent that will evaporate within the 2-hr. period.

(f) The dimensions listed in the following table for the various specimens shall be measured to the nearest 0.001 in. Dimensions not listed shall be measured within plus or minus  $\frac{1}{32}$  in.

Type of Specimen	Dimensions to be Measured to the Nearest 0.001 in.
Molded disk.....	thickness
Sheet.....	thickness
Rod.....	length and diameter
Tube.....	inside and outside diameter, and wall thickness

### Conditioning

4. Three specimens shall be weighed individually and then conditioned as follows:

(a) Specimens of materials whose water absorption value would be appreciably affected by temperatures in the neighborhood of 110 C., shall be dried in an oven for 24 hr. at  $50 \pm 3$  C., cooled in a desiccator, and immediately reweighed.

NOTE.—If a static charge interferes with the weighing, lightly rub the surface of the specimens with a grounded conductor.

(b) Specimens of materials, such as phenolic laminated plastics and other products whose water absorption value has been shown not to be appreciably affected by temperatures up to 110 C., shall be dried in an oven for 1 hr. at 105 to 110 C.

(c) When data for comparison with absorption values for other plastics are

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



desired, the specimens shall be conditioned in accordance with Paragraph (a).

#### Procedure

5. (a) *24-hr. Immersion.*—The conditioned specimens shall be placed in a container of distilled water maintained at a temperature of  $25 \pm 2$  C., and shall rest on edge and be entirely immersed. At the end of 24 hr., the specimens shall be removed from the water one at a time, all surface water wiped off with a dry cloth, and weighed immediately. If the specimen is  $\frac{1}{16}$  in. or less in thickness, it shall be put in a weighing bottle immediately after wiping and weighed in the bottle.

(b) *2-hr. Immersion.*—For all thicknesses of materials having a relatively high rate of absorption, and for thin specimens of other materials which may show a significant value in 2 hr., the specimens shall be tested as described in Paragraph (a) except that the time of immersion shall be reduced to 2 hr.

(c) *Repeated Immersion.*—A specimen may be weighed after 2-hr. immersion, replaced in the water, and weighed again after 24 hr.

NOTE.—In using this method the amount of water absorbed in 24 hr. is likely to be less than it would have been had the immersion not been interrupted.

(d) When data for comparison with absorption values for other plastics are desired, the 24-hr. immersion procedure described in Paragraph (a) shall be used.

#### Reconditioning

6. When materials are known or suspected to contain any appreciable amount of water-soluble ingredients, the specimens after immersion shall be reconditioned for the same time and temperature as used in the original drying period. They shall then be cooled in a desiccator and immediately reweighed. If the reconditioned weight

is lower than the conditioned weight, the difference shall be considered as water-soluble matter lost during the immersion test. For such materials, the water absorption value shall be taken as the sum of the increase in weight on immersion and of the weight of the water-soluble matter.

#### Calculation and Report

7. The report shall include the values for each specimen and the average for the three specimens as follows:

(1) Dimensions of the specimens before test, measured in accordance with Section 3 (f), and reported to the nearest 0.001 in.,

(2) Percentage loss in weight on initial drying, calculated on the basis of the original weight, and the conditioning time and temperature,

(3) The time of immersion,

(4) The percentage increase in weight during immersion calculated to nearest 0.01 per cent as follows:

Increase in weight, per cent =

$$\frac{\text{wet wt.} - \text{conditioned wt.}}{\text{conditioned wt.}} \times 100$$

(5) The percentage of soluble matter lost during immersion, if determined, calculated to nearest 0.01 per cent as follows (Note):

Soluble matter lost, per cent =

$$\frac{\text{conditioned wt.} - \text{reconditioned wt.}}{\text{conditioned wt.}} \times 100$$

NOTE.—When the weight on reconditioning the specimen after immersion in water exceeds the conditioned weight prior to immersion, report "none" under item (5).

(6) The percentage of water absorbed in 24 hr. (or in 2 hr.), which is the sum of the values in items (4) and (5), and

(7) Any observations as to warping, cracking, or change in appearance of the specimens.

# STANDARD DESCRIPTIVE NOMENCLATURE OF OBJECTS MADE FROM PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 675 - 45

ADOPTED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 675; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope.

1. (a) The purpose of this nomenclature is to provide simple terms, adequate to convey with maximum brevity and certainty, an accurate description of any visible characteristic of a plastic object which can be seen, but which cannot be expressed in numerical values. To simplify the nomenclature, no separate terms are provided to indicate variations in the prominence or extent of what otherwise would be the same characteristic (Note). To prevent misunderstandings which might arise through any choice of terms on too hasty assumptions as to the cause of a characteristic and to concentrate attention on a correct description of the visible characteristic, which is to be reported, the terms are made entirely descriptive in this nomenclature and without significance as to

the cause of what is described. Assumed causes should be stated separately.

NOTE.—It is very desirable to indicate such variations, but it is suggested that it be done by the addition of properly descriptive, commonly used adjectives, such as: pronounced, slight, faint, large, small, tiny, over-all, localized, numerous, few streaky, spotty, etc.

(b) This nomenclature may require the user to choose some different terms than have been used previously, and to place somewhat different interpretations on others, but it is hoped the ultimate advantages in so doing will outweigh any immediate disadvantages. Other terms which have been in use, although they seem superfluous, may not become obsolete. To assist in understanding such terms, they are listed under the heading "Terms Not Recommended" with their equivalence in recommended terms indicated. To those unfamiliar with the nomenclature relating to plastics, the following classification of terms may be helpful:

<sup>1</sup> Under the standardization procedure of the Society, this nomenclature is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to adoption as standard, this nomenclature was published as tentative from 1942 to 1943, being revised in 1943.

## CLASSIFICATION OF TERMS

*Terms Relating to Color Characteristics:*

Burned,  
Chalking,  
Discoloration  
Fading,  
Filler Specks,  
Hull,  
Lubricant-Bloom,  
Mold-Seam,  
Mottle,  
Resin-Streak,  
Segregation,  
Surface-Haze,  
Two-Tone, and  
Window.

*Terms Relating to Surface Characteristics:*

Burned,  
Chalking,  
Crater,  
Cut-Layers,  
Dry-Spot,  
Dull Surface,  
Frosting,  
Gloss,  
Lubricant-Bloom,  
Orange-Peel,  
Pimple,  
Plate-Mark,  
Pulled Surface,  
Resin-Streak,  
Scaly,  
Sheeter-Lines,  
Short,  
Surface-Haze,  
Thin-Spot, and  
Wrinkle.

*Terms Relating to Clarity:*

Clarity,  
Fish-Eye,  
Internal Haze,  
Optical Distortion,  
Ream,  
Slip-Plane,  
Striae,  
Translucent, and  
Internal Waviness.

*Terms Relating to Soundness or Structure:*

Bubble,  
Crack,

Crazing,  
Cut-Layers,  
Granular Structure,  
Mold-Seam,  
Open Bubble,  
Pinhole,  
Pit,  
Porosity,  
Pressure-Break,  
Pulled Surface,  
Resin-Pocket,  
Short,  
Weld Mark, and  
Window.

*Terms Relating to Shape:*

Blister,  
Dished,  
Domed,  
Sink-Mark,  
Waviness, and  
Warp.

## RECOMMENDED NOMENCLATURE

**Blister.**—Undesirable rounded elevation of the surface of a plastic, whose boundaries may be either more or less sharply defined, somewhat resembling in shape a blister on the human skin. A blister may burst and become flattened.

**Bubble.**—Internal, spherical void, or a trapped globule of air or other gas, especially in a transparent or translucent plastic.

**Burned.**—Showing evidence of thermal decomposition through some discoloration, distortion, or destruction of the surface of the plastic. See also *Discoloration*.

**Chalking.**—Dry, chalk-like appearance or deposit on the surface of a plastic.

**Clarity.**—Clearness.

**Crack.**—Actual fracture.

**Crater.**—Small, shallow, crater-like, surface imperfection.

**Crazing.**—Fine cracks which may extend in a network over or under the surface of or through a plastic.

**Cut-Layers.**—As applied to laminated plastics, a condition of the surface of machined or ground rods and tubes and of sanded sheets in which cut edges of the surface layer or lower laminations are revealed.

**Discoloration.**—Any change from an initial color possessed by a plastic; a lack of

- uniformity in color where color should be uniform over the whole area of a plastic object. In the latter sense, where they can be applied, use the more definite terms *Mottle*, *Segregation*, or *Two-Tone*.
- Dished.**—Showing a symmetrical distortion of a flat or curved section of a plastic object, so that as normally viewed, it appears concave or more concave. See also *Warp*.
- Domed.**—Showing a symmetrical distortion of a flat or curved section of a plastic object, so that as normally viewed, it appears convex or more convex. See also *Warp*.
- Dry-Spot.**—Area of incomplete surface film on laminated plastics; in laminated glass, an area over which the interlayer and the glass have not become bonded.
- Dull Surface.**—Lack of gloss or luster in the surface of a plastic, approaching a matte or sand-blasted finish.
- Fading.**—Any lightening of an initial color possessed by a plastic.
- Filler-Specks.**—Visible specks of a filler used, such as woodflour or asbestos, which stand out in color contrast against a background of plastic binder. It should be stated whether the specks are visible before or only after removal of the surface film.
- Fish-Eye.**—Small globular mass in a transparent or translucent plastic which has not disappeared or blended completely into the surrounding material, creating somewhat the appearance of a fish's eye.
- Foreign Matter.**—Particles of substance included in a plastic which seem foreign to its composition.
- Frosting.**—An apparently crystalline pattern on the surface of a plastic.
- Gloss.**—Brightness or luster of a body proceeding from a smooth surface.
- Granular Structure.**—Apparent incomplete fusion of and at least partial retention of their original form by the particles from which a plastic is formed, either within it or on its surface.
- Haze, Internal.**—Indefinite cloudy appearance within a transparent plastic.
- Haze, Surface.**—An indefinite cloud appearance on the surface of a plastic not describable by the terms *Chalking*, *Frosting*, or *Lubricant-Bloom*.
- Heat Mark.**—Extremely shallow, regular, or irregular depression or groove in the surface of a plastic having practically no depth (its area being very large compared with its depth) and visible because of a sharply defined rim or a roughened surface.
- Hull.**—Dark speck of foreign matter which appears to be in the fabric of fabric-base laminated sheet.
- Let-Go.**—An area in laminated glass over which an initial adhesion between interlayer and glass has been lost.
- Lubricant-Bloom.**—Irregular, cloudy, greasy exudation on the surface of a plastic.
- Mold-Seam.**—Line running lengthwise of a molded and machined, laminated rod or tube, differing in color or appearance from the general surface, and which appears to be a cut through the surface layer, or through the surface and under laminations.
- Mottle.**—Intentional or accidental mixture of colors or shades of a color giving a more or less distinct or complicated pattern of specks, spots or streaks of color.
- Open Bubble.**—Bubble which has broken partly through the surface of a plastic. See also *Bubble*, *Pinhole*.
- Optical Distortion.**—An apparent distortion of anything viewed through a transparent plastic, caused by the nonuniform optical character of the plastic, and not by its shape.
- Orange-Peel.**—Uneven surface somewhat resembling an orange peel.
- Pimple.**—Undesirable small sharp or conical elevation on the surface of a plastic whose form resembles a pimple in the common meaning. See also *Blister*.
- Pinhole.**—Tiny but apparently very deep hole in the surface of, or through a thin section of plastic, which usually is round and has a rounded edge.
- Pit.**—Small regular or irregular hole in the surface of a plastic, usually having somewhat like diameter and depth, and often dull or rough at the bottom. See also *Pinhole*, *Short*.

**Plate-Mark.**—A duplication in a laminated sheet of finish lines or an imperfection in the surface of the pressing plate.

**Porosity.**—Presence of numerous visible voids.

**Pressure-Break.**—An apparent break in one or more outer sheets of the paper, fabric, or other base of a laminated plastic, visible through the surface layer of resin which covers it.

**Pulled Surface.**—Imperfections in the surface of a laminated plastic ranging from a slight breaking or lifting of its surface in spots to pronounced separation of its surface from its body.

**Ream.**—Layers of unhomogeneous material parallel to the surface in a transparent or translucent plastic.

**Resin-Pocket.**—An apparent accumulation of excess resin in a small localized area between laminations in laminated plastics, visible on cut edges or molded surfaces.

**Resin-Streak.**—Streak of what appears to be excess resin on the surface of a laminated plastic.

**Scaly.**—Showing a flaked surface appearance.

**Scratch.**—Shallow mark, groove, furrow, or channel.

**Segregation.**—A close succession of parallel, rather narrow and sharply defined, wavy lines of color on the surface of a plastic differing in shade from surrounding areas, and creating the impression that components of the plastic have separated.

**Sheeter Lines.**—Parallel scratches or projecting ridges distributed over considerable area of a plastic sheet such as might be produced during a slicing operation.

**Short.**—Incompletely filled-out condition in a molded material which may be quite obvious, or only evident through absence of surface film in tiny or larger areas, or as lighter, unfused particles of material show through a covering surface film, accompanied possibly by peculiar thin-skinned blisters. With thermosetting materials, it is important to note whether the short occurs only after a period of prewarming the mold charge, or as a result of a delay in closing the mold.

**Sink-Mark.**—Dimple-like depression in the surface of a molding where it has retracted from the mold, and which has well rounded edges, and in which there is no absence of surface film.

**Slip-Plane.**—Plane within transparent plastic visible in reflected light because of poor welding and shrinkage on cooling.

**Striae.**—Surface or internal thread-like in homogeneities in transparent plastic.

**Thin-Spot.**—An unpolished or poorly finished area on an otherwise polished sheet of plastic material.

**Translucent.**—Allowing the passage of some light, but not a clear view of any object.

**Two-Tone.**—Two shades of its nominal color more or less entirely covering adjacent areas on a molding with a more or less sharp line of demarcation between.

**Warp.**—Dimensional distortion in a plastic object after molding or other fabrication. See also *Dished*, *Domed*.

**Waviness, Surface.**—Wave-like unevenness, or out-of-plane, in the surface of a plastic.

**Waviness, Internal.**—An appearance of waviness seen in a transparent plastic.

**Weld-Mark.**—Mark formed by the incomplete union of two or more streams of plastic flowing together.

**Window.**—A tiny, colorless, transparent area or speck in a sheet of colored or opaque plastic which looks like a hole when the sheet is held to the light.

**Wrinkle.**—A surface imperfection in laminated plastics that has the appearance of a crease or wrinkle in one or more outer sheets of the paper, fabric, or other base which has been pressed in.

#### TERMS NOT RECOMMENDED

**Bloom.**—Use *Lubricant-Bloom*.

**Blush.**—Used loosely as having the various meanings of *Chalking*, *Frosting*, or *Lubricant-Bloom*, use definitions for these terms.

**Boil.**—Small bubble  $\frac{1}{32}$  to  $\frac{3}{32}$  in. in diameter. Use *Bubble* and state size if desirable to do so.

**Bulging.**—Used either to denote a very large blister or as a synonym for *Doming*. Use *Blister* or *Domed*.

**Cat's Eye.**—Use *Fish-Eye*.

**Chicken-Skin.**—Use *Orange-Peel*.

*Cold*.—Dull surface apparently attributed to a cold mold. Use *Dull Surface*.

*Cord*.—Use *Striae*.

*Crush*.—Use *Pressure-Break*.

*Densification*.—Accidental areas of lesser translucency in a translucent plastic not due to its shape. Describe as lacking translucency or opaque in certain areas, etc.

*Dimple*.—Use *Sink-Mark*.

*Dog-Skin*.—Use *Orange-Peel*.

*Dry-Area*.—Use *Dry-Spot*.

*Flow-Lines*.—Used loosely with the same meaning as *Striae*, or *Weld-Mark* (use definitions for these terms) or to denote any streakiness taken to indicate the path followed by a plastic.

*Fog*.—Use *Haze*.

*Gas-Mark*.—Accidental areas of lesser trans-

lucency in a translucent plastic not due to its shape. Describe as lacking translucency or opaque in certain areas, etc.

*Gas-Pocket*.—Use *Heat-Mark*.

*Grease-Mark*.—Use *Lubricant-Bloom*.

*Inverted Blister*.—Use *Sink-Mark*.

*Low-Spot*.—Use *Thin-Spot*.

*Pebble*.—Use *Orange-Peel*.

*Piping*.—Use *Sink-Mark*.

*Pock-Mark*.—Use *Pit*.

*Precure*.—A *Short* which occurs after preheating of a mold charge or a delay in closing a mold. See *Short*.

*Seed*.—Small bubble  $\frac{1}{8}$  in. in diameter. Use *Bubble*.

*Skip*.—Use *Thin-Spot*.

*String*.—Use *Striae*.

*Tear-Drop*.—Use *Fish-Eye*.

*Unconverted-Spot*.—Use *Window*.

## *Standard Methods of* SAMPLE PREPARATION FOR PHYSICAL TESTING OF RUBBER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 15 - 41

ADOPTED, 1939; REVISED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 15; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These general methods are intended to apply to soft vulcanized rubber compounds such as are used for the lining and cover of hose and belting, for carcass and tread of tires, and for a wide variety of molded or extruded rubber products. They are not applicable without modification to material ordinarily classed as hard rubber.

(b) Only instructions for the preparation of test specimens of rubber products and the general requirements for approved testing are included in these general methods. Standard methods for individual test procedures are provided separately or in specifications covering a particular material.

(c) In case of conflict between the provisions of these methods and those of the individual test procedures or specifications for a particular material, the latter shall take precedence.

### Place of Test

2. Tests shall be made at the place of manufacture, unless otherwise specified. The manufacturer shall provide the requisite facilities for conducting tests, including labor and equipment.

### Sampling

3. (a) Samples shall be obtained, if possible, from the product to be tested and in such case shall be selected in accordance with the requirements of the specifications covering the particular material.

(b) In case it is not practicable to obtain suitable test specimens from the finished article, the manufacturer shall, upon request, furnish pieces approximately 6 by 6 by 0.075 in. which he guarantees to be of the same material and equivalent cure as that used in the article.

### Preparing Samples of Vulcanized Rubber

4. In case samples of vulcanized rubber are to be prepared for purpose of tests from the compounding ingredients,

<sup>1</sup> Under the standardization procedure of the Society these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to their present adoption as standard, these methods were published as standard from 1915 to 1935, being revised in 1924 and 1932, but were withdrawn and republished as tentative from 1935 to 1939, being revised in 1938.

the preparation procedure<sup>3</sup> described in the following Paragraphs (a) to (e) shall be used:

(a) All ingredients shall be accurately weighed to within 0.25 per cent of the weight specified. The final weight of the mixed batch and the sum of the weights of all ingredients shall not differ by an amount exceeding 0.6 per cent for a compounded batch or 0.3 per cent for pure gum or master batched stocks.

(b) Mixing of the rubber compound shall be done on a laboratory rubber mill with rolls 12 in. in width by 6 in. in outside diameter having a working distance between the guides of  $10\frac{1}{2}$  in. The speed of the slow roll shall be 24 rpm. and the gear ratio 1.4, or as near that figure as is compatible with good engineering practice. Sufficient cooling water shall be circulated through the mill rolls to maintain a roll temperature of approximately 158 F. (70 C.).

(c) The rubber portion of the batch shall be broken down on a warm mill with mill opening of 0.055 in. until it runs smooth. The mill then shall be adjusted to the required opening in conformity with the following:

Volume of Batch, ml.	Distance Between Rolls, in.
1200.....	0.170
1100.....	0.160
1000.....	0.145
900.....	0.130
800.....	0.120
700.....	0.110
600.....	0.100
500.....	0.085
400.....	0.070
300.....	0.055

The compounding ingredients shall be next added to the rubber as rapidly as possible in the following order:

- (1) Accelerators and antioxidants
- (2) Carbon black
- (3) Fillers
- (4) Softeners
- (5) Sulfur

After the ingredients are all incorporated, the batch shall be cut six times, alternating first from one side and then the other. This is to be interpreted as cutting two-thirds of the way across the roll and holding the rubber until the bank just disappears. After the six cuts are completed, the batch shall be cut across and rolled six times with the roll inserted endwise each time until the last when it shall be placed in the rolls lengthwise. Then, as soon as the bank is well balanced, the mill setting shall be

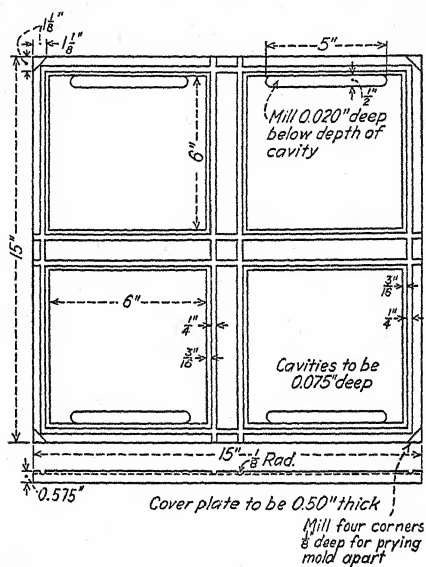


FIG. 1.—Design for Four Cavity Mold.

adjusted to give a sheet approximately 0.085 in. in thickness, after cooling and the batch is cut from the rolls.

(d) The batch shall be cooled to room temperature on a flat surface and cut into slabs  $\frac{1}{8}$  in. less in width and length than the corresponding dimensions of the mold cavity. Direction of grain of the rubber shall be marked on each slab. The unvulcanized stock shall be aged 18 to 24 hr. before cure.

NOTE.—It is desirable, when possible, that the aging be in subdued light in air having a relative humidity of 45 per cent at 82 F. (28 C.).

<sup>3</sup> This procedure is substantially in agreement with that of the Physical Testing Committee, Division of Rubber Chemistry, Am. Chemical Soc.



(e) The test slabs shall be vulcanized in a mold similar to that shown in Fig. 1, by being heated at a specified temperature for a given time while held under molding pressure. For this purpose a hydraulic press with heated platens is convenient. The press shall give an intensity of pressure on the mold of not less than 500 psi. The platens shall be of such dimensions that no portion of the slab while curing is nearer than 3 in.

The time of cure shall be that between the moment the hydraulic pressure reaches its maximum and the moment of release of the hydraulic pressure. The mold shall be brought to the curing temperature before inserting the uncured stock by being placed in the closed press for a period of not less than 20 min. The minimum possible time shall elapse after the stock is placed in the hot mold until the full hydraulic pres-

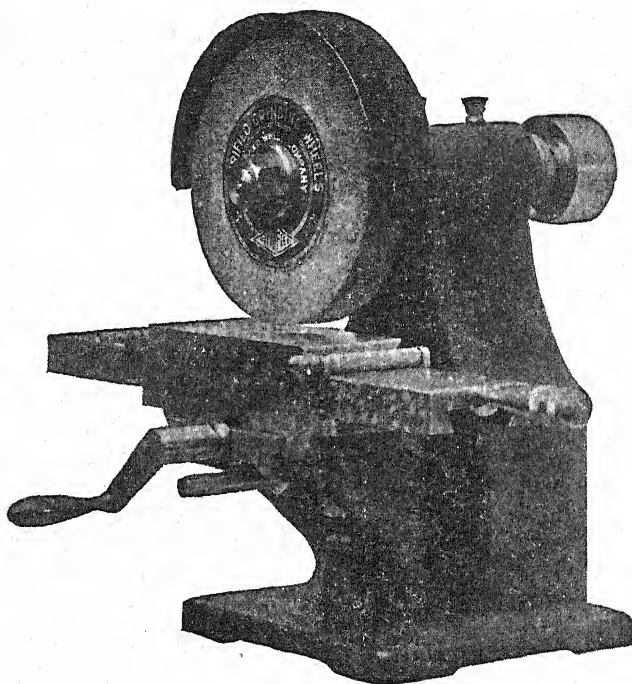


FIG. 2.—Grinder for Buffing Samples.

to the edge of the platen. In order to avoid cool spots in the platens due to condensed moisture, presses of the bored platen type are recommended. If platens of the chamber type are used, particular attention must be paid to insuring good drainage by placing the steam outlet slightly below the steam chamber. In either case, the actual platen temperatures shall be verified by means of thermocouples in the mold or other suitable temperature measuring device.

sure is applied. The slabs shall be removed from the mold immediately after the conclusion of the cure and cooled in water for 10 min. after which they shall be wiped dry and reserved for test.

#### Age of Samples

5. All tests of rubber shall be made using specimens which have aged at least 24 hr. after vulcanization. Tests of rubber made more than 60 days after vulcanization shall not be considered as

evaluating the original qualities of the material.

NOTE.—Finished rubber products are not in a completely stable condition as regards physical properties. Immediately after cure, the rate of change is rapid. During the first 12 to 24 hr. the rate becomes progressively less until a minimum is reached. Very slow change continues, however, throughout the useful life of the article and is accelerated when the rubber is exposed to heat, light, and air.

### Condition of Samples

6. Samples and specimens for test shall not be heated, immersed in water, nor subjected to any mechanical or chemical treatment prior to test except as specifically prescribed herein or in the standard test method. Any such treatment may definitely change the results of test. Particular attention is necessary to avoid previous stretching through inadvertent handling.

### Temperature of Test

7. All tests of rubber shall be made at a room temperature between 70 and 90 F. (21 and 32 C.). Test specimens shall be conditioned at the test temperature for at least 30 min. just prior to test. The actual test temperature shall be recorded in all cases. Close comparisons should not be made between test results obtained under temperature conditions differing by more than 10 F. (5.5 C.).

### Separation of Rubber

8. (a) When the tests to be made require prior separation of rubber from cotton fabric or other material, the separation shall be performed without the use of gasoline, if possible, and without rupture or excessive stretching. To avoid unnecessary stretching, it is desirable to cut the material into strips slightly wider than a test specimen. The separation shall be made gradually

and a little at a time while the rubber is gripped near the point of separation.

(b) If it is necessary to employ gasoline to effect the separation, it shall be straight-run gasoline of the quality known as aviation grade and shall have the following characteristics:

Specific gravity 60/60 F. .... 0.685 to 0.705  
Distillation limits:

Initial boiling point. ....	100 to 120 F.
10 per cent evaporated. ....	128 to 143 F.
20 per cent evaporated. ....	140 to 155 F.
30 per cent evaporated. ....	150 to 164 F.
40 per cent evaporated. ....	156 to 174 F.
50 per cent evaporated. ....	164 to 184 F.
60 per cent evaporated. ....	173 to 194 F.
70 per cent evaporated. ....	182 to 207 F.
80 per cent evaporated. ....	193 to 223 F.
90 per cent evaporated. ....	209 to 245 F.
95 per cent evaporated. ....	224 to 260 F.
Dry. ....	250 to 286 F.
Recovery, min. ....	97 per cent

The gasoline shall contain no lead compounds and shall not leave an appreciable amount of oily residue when evaporated.

After use of gasoline, the rubber shall be placed so as to permit free evaporation from all parts of its surface and shall be allowed to rest at least 1 hr. before being tested.

### Buffing

9. Samples of rubber shall be buffed when necessary to remove unevenness of surface which would interfere with an accurate measurement of thickness. Any unevenness of surface such as fabric impression or corrugations caused by contact with fabric components or with cloth wraps used for curing shall be removed by careful grinding, taking care to prevent excessive heating of the rubber samples. The grinder shall be equipped with an abrasive wheel of approximately No. 30 grit, 5 in. in diameter, revolving at a speed of 2500 to 3500 rpm. and shall be provided with a slow feed. Suitable buffing equipment is shown in Figs. 2 and 3. In removing unevenness of surface, buffing should not be carried beyond the point at which the unevenness in gage just

disappears except in the case of thick specimens where it is necessary to reduce the thickness for tension tests. The speed and cut shall be such as to result in a smooth uniform buffed surface with

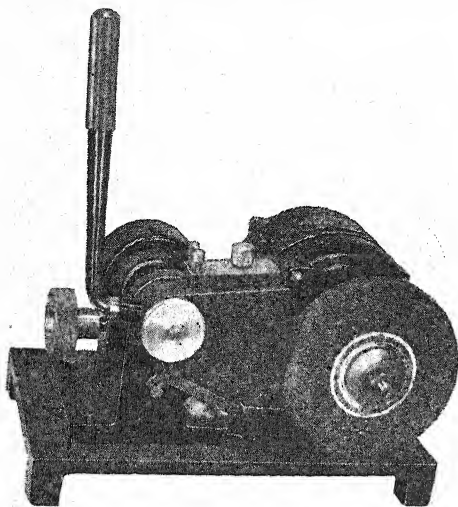


FIG. 3.—Grinder for Buffing Samples.

a minimum of sample heating. When necessary to reduce the thickness of a sample for preparation of test specimens it may be desirable to slice the rubber approximately to the desired thickness and finish by buffing. Samples for preparation of tension specimens shall be

buffed in strip form prior to dieing out the specimens. At least 30 min. shall elapse between the buffing and testing of a specimen except that when minimum requirements are established, as in testing for compliance with a particular specification, the rest period may be reduced to 10 min.

NOTE.—Specimens with buffed surfaces instead of mold finish usually give slightly lower tensile strength test results because of the tendency for surface roughness to favor premature breaking of the specimens.

### Methods of Testing

10. All tests of rubber parts shall be carried out in accordance with the requirements of the American Society for Testing Materials standard specifications for the material or the A.S.T.M. standard test method for determining the specific physical property required.

### Retests

11. Any lot represented by a sample which fails in one or more tests may be retested. For this purpose two additional samples shall be selected. Failure of either in any respect shall be cause for rejection.

## *Standard Methods of* TENSION TESTING OF VULCANIZED RUBBER<sup>1</sup>



A.S.T.M. Designation: D 412 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 412; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods describe the procedures for determining stress-strain properties, tensile strength, ultimate elongation, and permanent set of the usual grades of soft vulcanized rubber such as tire carcass and tread compounds or those used in most mechanical rubber goods. They do not apply to the testing of material ordinarily classed as hard rubber.

### Description of Terms

2. (a) *Tensile Strength* is the tension load per unit cross-sectional area required to break a test specimen of the type and in the manner herein described.

NOTE.—Since rubber when so stressed usually stretches to a high degree with accompanying substantial reduction in cross-sectional area, and since precise measurement of the dimensions at the moment of breaking is not possible, the calculation of intensity of load is referred in all cases to the original cross-section.

(b) *Elongation* is the extension produced by a tension load in a section of a

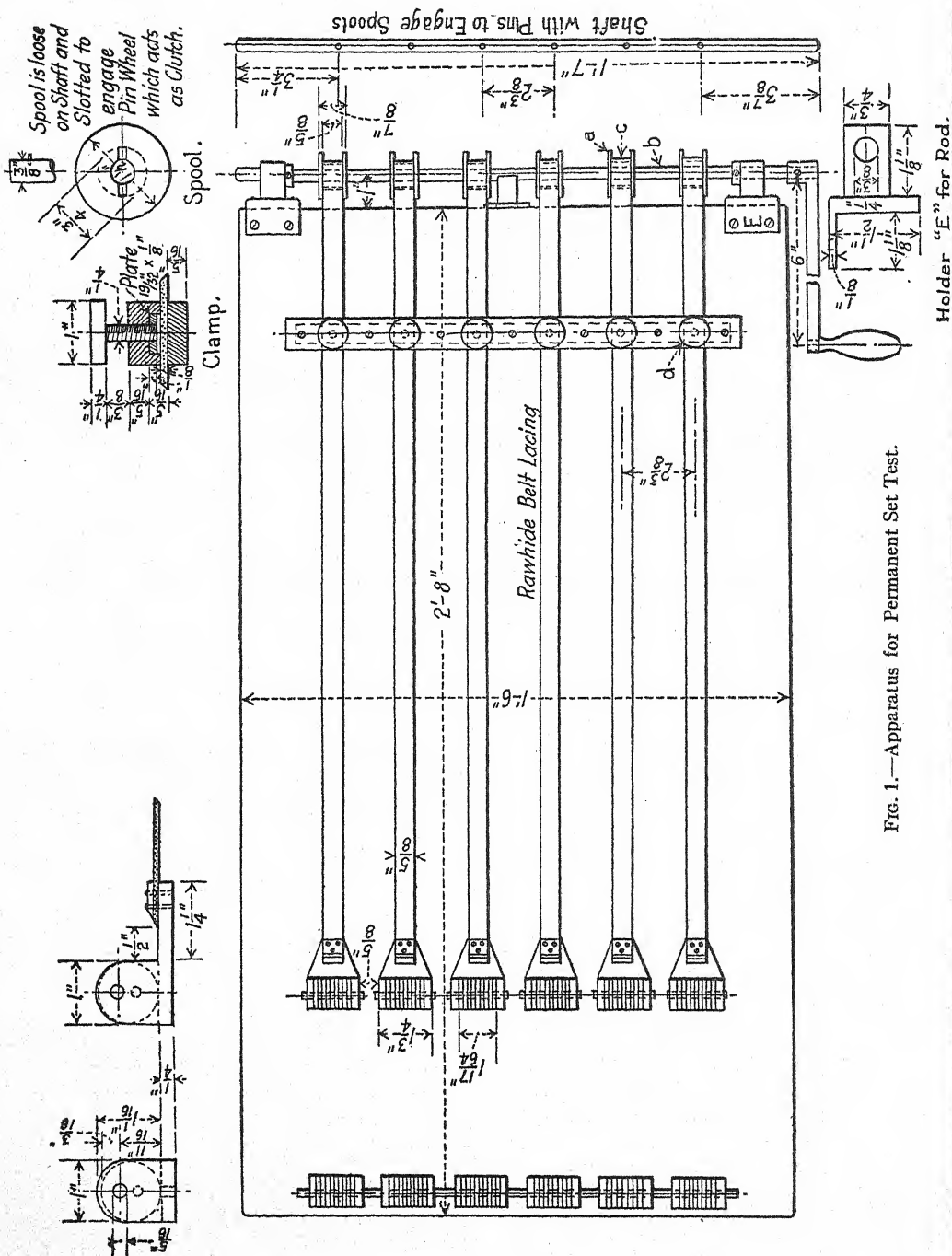
test specimen between gage marks placed on it, and is expressed as a percentage of the original distance between the marks. Ultimate elongation is the elongation at the moment of rupture. Elongation is also correctly designated by the term "strain."

(c) *Tensile Stress* is the intensity of load referred to the original cross-section at the corresponding stated percentage of elongation which it produces. It is expressed in pounds of tension load per square inch at the particular elongation, for example, 1000 psi. at 500 per cent elongation, and is often designated by the term "modulus." It is used as an index of stiffness.

(d) *Permanent Set* refers to the extension remaining after a test specimen has been stretched and held at a specified elongation for a given period of time, relieved of load, and allowed to recover during a specified interval of rest as described in these methods. It is expressed as a percentage of the original length and is used as an index of elasticity.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1935 to 1941, being revised in 1936, 1939, 1940, and 1941.



### Tension Test Apparatus

3. Tensile strength, elongation, and stress-strain tests shall be made on a power-driven apparatus, preferably of the inclination-balance or pendulum type. A spring-balance type of apparatus may be used if provided with a device which will indicate the actual maximum load at which rupture took place and if provision is made to prevent recoil of the spring. The machine shall fulfill the following requirements:

(a) The applied tension as indicated by a dial or scale shall be accurate within plus or minus 1 per cent.

(b) The indicator shall remain at the point of maximum load after rupture of the test specimen.

(c) The grips which hold the specimen in the testing machine shall be of a type which tightens automatically and exerts a uniform pressure across the gripping surface proportional to the applied tension so as to avoid uneven slipping and to insure failure of the test specimen in its constricted portion.

(d) The rate of travel of the power actuated grip shall be 20 in. per min. and shall be uniform at all times.

(e) Provision shall be made for measuring the elongation by a scale graduated to 0.1 in. and suitably attached to measuring trammel points or held back of the test specimen.

(f) The testing machine shall be calibrated in accordance with the provisions of Section 4.

(g) The tension testing machine may be equipped with a dynamometer head of the compensating type for convenience in eliminating calculations.

(h) The testing machine used for a given test specimen shall be of such capacity that the maximum load required to break the specimen shall not exceed 85 nor be less than 15 per cent of the rated capacity. For tests of most rub-

ber compounds using one of the test specimens described in these methods a machine of 150-lb. maximum capacity will be satisfactory. For general use in tension testing of rubber, possible separation of the grips of at least 30 in. shall be provided.

### Calibration of Tension Testing Machines

4. Tension testing machines shall be calibrated by dead-weights applied in an ascending order while the machine is otherwise arranged in an entirely similar manner to that used when testing material. In each case, when additional calibrating weight is added the weight lever of the machine shall be moved to zero position and allowed to swing slowly upwards to the point where it will go no further. The dial of the machine should then clearly indicate the amount of the weight applied.

### Set Test Apparatus

5. Permanent set tests may be carried out on the power driven tension machine described in Section 3; or the special apparatus illustrated in Fig. 1 may be employed.

### Preparation of Sample

6. Except as may be otherwise specified in these methods, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials<sup>3</sup> shall be complied with and are made a part of these methods.

### Die for Test Specimens

7. Unless otherwise prescribed in the specifications for the material being tested, the specimens for tension tests shall be stamped out with a steel die constructed substantially in accordance with

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Fig. 2. The dies shall be kept sharp and free from nicks to avoid leaving ragged edges on the rubber. Cutting may be facilitated by wetting the rubber surface and the cutting edges of the die. The rubber shall be rested on a smooth, slightly yielding surface which will not injure the blade. Lightweight cardboard or a piece of leather belting is suit-

the grain, and it is assumed that unless otherwise specified all test specimens are to be prepared in this manner. Where grain effects are significant and are to be evaluated, two sets of test specimens should be cut at right angles to each other.

### Test Specimens

8. (a) The test specimens shall conform in shape to die A, B, C, or D as

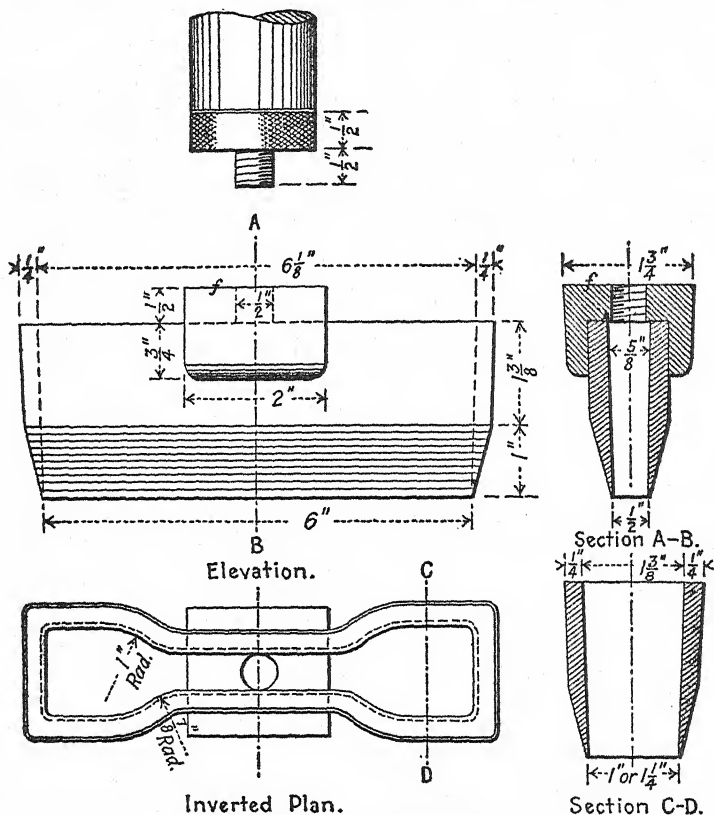


FIG. 2.—Die for  $\frac{1}{2}$  by 2-in. Specimens.

able. Care shall be taken that the cut edges are perpendicular to the other surfaces of the specimen and have a minimum of concavity.

NOTE.—It is a well known fact that vulcanized rubber exhibits a grain effect which influences its physical properties. In the case of heavily loaded compounds the effect may produce a pronounced difference in physical properties. Laboratory practice is to test with

shown in Fig. 3, and shall not vary in width in the reduced section by more than plus or minus 0.001 in. from the dimension shown. In no case shall the thickness of the test specimen exceed  $\frac{1}{8}$  in.

(b) Die B or die C shall be used in all cases where the dimensions of the sample and the capacity of the testing machine permit except when otherwise specified



in detailed specifications or test methods covering a particular product which shall take precedence. Die B shall be used only in preparing specimens from compounds having high filler content. When the dimensions or form of a sample are such as to preclude the preparation of adequate specimens using die B

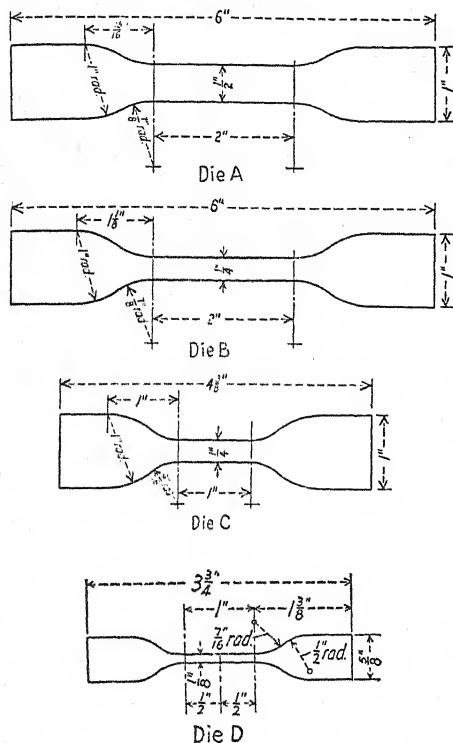


FIG. 3.—Tensile Strength and Elongation Test Specimens.

or die C, die D shall be used. In all cases in which the sample and the testing machine limitations permit a choice in die shapes, the die giving the higher test values shall be used as such higher results will be nearer the true value of the material.

(c) Two parallel gage lines for use in determining elongation shall be marked on the reduced section perpendicular to the longitudinal axis of the specimen, one

on each side of the center and  $\frac{1}{2}$  in. or 1 in. therefrom as required. The marks shall be as narrow as possible consistent with legibility. Use of a stamp with parallel steel blades is recommended but care shall be taken not to injure the rubber.

(d) The thickness of the specimen shall be measured with a micrometer graduated to 0.001 in. having a presser foot  $0.25 \pm 0.01$  in. in diameter exerting a total force of  $3.0 \pm 0.1$  oz. The load shall be applied by means of a weight.

(e) The minimum thickness and width of the specimen between the gage marks shall be taken in calculating the area used in determining the tensile strength of the specimen. In the case of specimens prepared with die D, the measurement of thickness shall be taken with the center of the micrometer foot coinciding with the longitudinal center line of the specimen so that there is an equal overlap of the foot on either side of the specimen.

#### Number of Test Specimens

9. (a) Three specimens per sample shall be tested for determinations of tensile strength, elongation, and stress-strain properties, except as noted in Paragraph (b). Results on specimens which break outside of the straight reduced section or which are found to be imperfect shall be discarded and retests shall be made. In case the ultimate tensile strengths fail to check within 10 per cent of the highest value obtained, additional specimens shall be tested until two or more such check results are obtained. The final values shall be the averages of the check tests, discarding the determinations on samples which are outside of the above limit.

(b) When minimum requirements are established, as in testing for compliance with a particular specification, only one test shall be made for tensile strength,



ultimate elongation, or stress-strain relations. If the results are below the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to conform to the specifications.

(c) Two specimens per sample shall be tested for permanent set and the results shall not differ by more than 25 per cent of the percentage values obtained. In case of greater variation than specified, additional specimens shall be tested. The final value shall be the average of the check tests. Results on specimens found to be imperfect shall be discarded and retests shall be made.

#### Procedure

10. (a) *Tensile Strength and Ultimate Elongation*.—The test specimen shall be placed in the grips of the testing machine, using care to adjust them symmetrically in order that the tension shall be distributed uniformly over the cross-section. The grips shall be separated at the specified rate of speed until rupture takes place, and during separation the distance between the gage marks on the specimen shall be noted continuously so that at the instant of rupture the distance is known within 0.1 in. This value shall be recorded together with the original distance between the gage marks from which data the ultimate elongation may be calculated. After rupture of the specimen, the breaking load in pounds shall be noted from the dial or scale and recorded together with the original minimum width and thickness of the specimen so that the tensile strength may be calculated.

(b) *Stress-Strain Properties*.—The procedure shall be the same as described in Paragraph (a) except that the load and extension shall be recorded at equal in-

crements of 100 per cent elongation as well as at rupture. Use of an autographic or spark recorder for registering observed loads is recommended.

(c) *Permanent Set*.—The test specimen shall be placed in the grips as described in Paragraph (a) and the grips separated at the machine speed or by hand at a uniform rate of speed such as to require approximately 15 sec. to reach the specified elongation. The specimen shall be held in this stretched position for 10 min., released immediately but without being allowed to snap back, and allowed to rest for 10 min. The distance between the gage marks shall then be measured to the nearest 0.01 in. and recorded together with the original distance between the gage marks. The difference is the measurement of the permanent set. If no specified elongation is given for the permanent set determination, the test specimen shall be stretched an amount equal to three-fourths of the ultimate elongation determined as in Paragraph (a).

#### Calculations

11. (a) *Tensile Strength* shall be calculated by dividing the breaking load in pounds by the original area of the cross-section of the test specimen in square inches and shall be expressed in pounds per square inch.

(b) *Ultimate Elongation* shall be calculated by subtracting the original distance between gage marks in inches from the total distance between the marks at the time of rupture as measured in Section 10 (a) and expressing this difference as a percentage of the original distance as follows:

$$\text{Ultimate elongation, per cent} = \frac{D-G}{G} \times 100$$

where:

$D$  = distance at break, and  
 $G$  = original distance.

(c) *Stress-Strain Properties*.—Tensile stresses shall be calculated as in the case of tensile strength except that the observed loads shall be used in place of the breaking load. Elongation or strain shall be calculated as in the case of ultimate elongation except that the observed distance between the gage marks at each recorded extension shall be used in place of the distance at break.

(d) *Permanent Set* shall be calculated as a percentage of the original distance between the gage marks by taking the difference between the gage mark distance after the rest period and the original distance as described in Section 10 (c), multiplying by 100 and dividing the product by the original distance.

#### Report

12. The report shall include the following:

(1) The results calculated in accordance with Section 11,

(2) All observed and recorded data on which the calculations are based,

(3) The date of vulcanization of the rubber, if known,

(4) The date of test,

(5) The temperature of the test room,

(6) The type of testing machine used, and

(7) The dimensions of the die used in preparing the test specimens.

*Standard Method of Test for*  
**ACCELERATED AGING OF VULCANIZED RUBBER BY THE  
OXYGEN-PRESSURE METHOD<sup>1</sup>**



**A.S.T.M. Designation: D 572 - 42**

ADOPTED, 1940; REVISED, 1941, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 572; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Scope**

1. This method is intended for use in estimating the relative resistance of vulcanized rubber to age deterioration. No direct correlation between this accelerated test and natural life of rubber is given or implied. Since the rate at which deterioration proceeds during the normal life of rubber varies widely depending on conditions of exposure to heat, light, and air and on the composition and state of cure of the rubber, this accelerated test is comparative only and must be evaluated against the performance of rubber compounds of which both the natural and accelerated aging characteristics are known.

**Nature of Test**

2. (a) This aging test of rubber consists of subjecting test specimens having

previously determined physical properties to controlled deteriorating influences for known periods, after which the physical properties are again measured and the changes noted. In this method the test involves exposure of specimens to an elevated temperature and elevated oxygen pressure. This test is designated as an oxygen-pressure-chamber aging test. It does not include exposure to light.

(b) In this aging test, the physical properties used to measure the deterioration of the rubber, in addition to visual and manual inspection, are tensile strength and ultimate elongation or stress-strain relationship. Except as otherwise specified in this method, the determination of these properties before and after aging shall be carried out in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials.<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> This method formerly comprised the oxygen-pressure test procedure in the former Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (D 428-36 T), which was published as tentative from 1935 to 1940, being revised in 1936.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### General Methods

3. (a) Except as otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials<sup>3</sup> shall be complied with and are made a part of this method.

(b) In case of conflict between the provisions of this method and those of detailed specifications or methods of test for a particular material, the latter shall take precedence.

### Apparatus

4. The oxygen-pressure chamber shall consist of a metal vessel designed to retain an internal atmosphere of oxygen gas under pressure, with provisions for placing rubber specimens within it and subjecting the whole to controlled uniform temperature. Two suggested de-

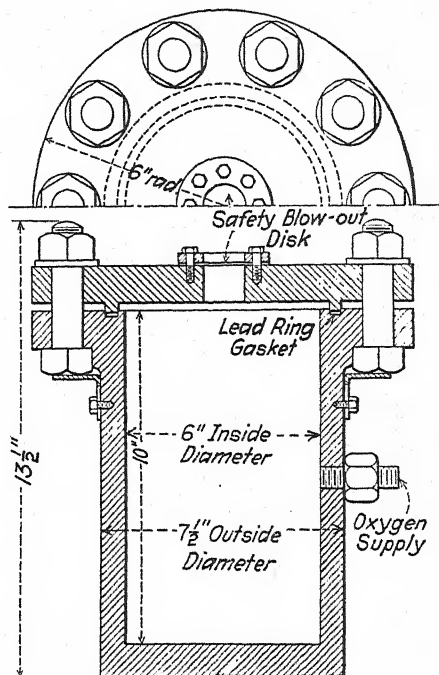


FIG. 1.—Oxygen Pressure Aging Chamber, Nonjacketed for Water or Air Bath.

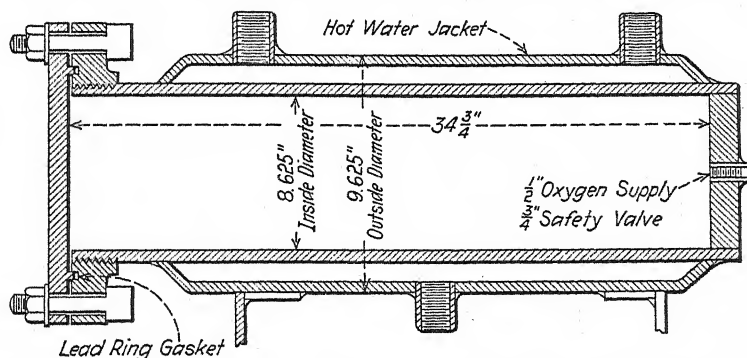


FIG. 2.—Oxygen Pressure Aging Chamber, Hot-Water Jacketed.

signs which are suitable are shown in Figs. 1 and 2. The equipment shall conform to the following requirements:

(a) The size of the chamber shall be optional but shall be such that the specimens may be suspended therein vertically without undue crowding and

without touching each other or the sides of the chamber.

(b) The source of heat is optional but shall be located outside of the aging chamber proper.

(c) The heating medium is optional but water is preferred because of more

rapid heat transfer. When the design shown in Fig. 1 is used, it is convenient to employ for the heating a calorimeter water bath in which the entire chamber is immersed. If air is used, the following special precautions shall be taken in order that accurate, uniform heating is obtained in all parts of the aging chamber:

(1) The heated air shall be thoroughly circulated around the vessel in the oven housing by means of mechanical agitation, and

(2) Baffles shall be used as required to prevent local overheating and dead spots.

(d) Automatic temperature control of the heating medium by means of thermostatic regulation shall be used. If air is used as the heating medium, the preferred location of the temperature control is adjacent to the recording thermometer.

(e) A recording thermometer located in the heating medium shall be provided to record the actual temperature. Preferably, the thermometer bulb shall be close to the wall of the pressure chamber but not touching it. If air is used as the heating medium, an actual check of temperature shall be made by means of maximum reading thermometers placed in various parts of the oven housing to verify the uniformity of heating.

(f) Positive, rapid, and complete circulation of the heating medium shall be maintained so as to assure accurate, uniform heating.

(g) The pressure chamber shall be equipped with a reliable safety valve set for release at 500 psi. pressure.

**NOTE: Caution.**—Adequate safety provisions are important when heating oxidizable organic materials in oxygen since the rate of reaction may become very rapid in some cases, particularly if large surface area is exposed, and very high pressures may be developed.

## Test Specimens

5. (a) Dumbbell-shaped test specimens, prepared as described in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials<sup>3</sup> shall be considered standard. Their form shall be such that no mechanical, chemical, or heat treatment will be required after aging. If buffing is necessary, it shall be performed prior to the aging cycle.

(b) The dimensions of the test specimens for calculating the physical properties shall be measured prior to starting the aging cycle. Care shall be taken that the material used for marking the gage lines on the specimens is not detrimental to the rubber during aging exposure, causing the specimens to break at the marks prematurely during test. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other.

## Number of Test Specimens

6. (a) At least three specimens shall be used to determine the average original physical properties of the rubber and also three or more specimens of the same material for each interval of the aging test.

(b) When minimum requirements are established, one test shall be made for tensile strength and elongation. If the results are below the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to meet the specifications.

## Tests of Unaged Specimens

7. (a) The stress-strain properties or tensile strength and ultimate elongation

of the original unaged specimens shall be determined within 24 hr. of the start of the aging period. Results on specimens which break outside of the straight reduced section or which are found to be imperfect shall be discarded, and retests shall be made.

(b) When rubber compounds are to be tested for the purpose of determining compliance with a specification, it shall be permissible to determine the original properties required in Paragraph (a) simultaneously with the determination of the values after the first aging period even though the elapsed time exceeds 24 hr.

#### Procedure for Accelerated Aging

8. (a) The specimens for aging shall be placed in the aging chamber after it has been preheated to the operating temperature. It is recommended that not more than 2.0 g. of actual rubber or oxidizable substance per cubic inch of capacity be placed in the chamber. Simultaneous aging of a mixed group of different compounds shall be avoided if possible. For instance, high-sulfur compounds should not be aged with low-sulfur compounds and those containing antioxidants should not be aged with those having no age-resistors. Some migration is known to occur. When starting a test, the air shall be flushed out of the oxygen-pressure chamber by releasing the oxygen pressure and refilling, and the chamber shall be checked to make certain the apparatus does not leak.

(b) The operating temperature shall be  $70 \pm 1$  C. determined by measuring the temperature of the heating medium which shall be considered the same as that of the pressure chamber.

(c) The pressure of oxygen supplied to the aging chamber shall be  $300 \pm 10$

psi. as measured by a calibrated pressure gage.

(d) The aging interval shall start at the time the specimens are placed in the heated chamber and shall continue for a measured time interval. The selection of suitable intervals of aging will depend on the rate of deterioration of the particular material being tested. Time intervals frequently used are 24, 48, 72, and 96 hr.

(e) The aging intervals used shall be such that the deterioration will not be so great as to prevent determination of the final physical properties. In experimental work, it is desirable to use a range of periods, while for routine tests of known materials fewer intervals may be employed.

(f) At the termination of the aging interval, the specimens shall be removed from the aging chamber, cooled to room temperature on a flat surface and allowed to rest not less than 16 hr. nor more than 48 hr. before determination of the physical properties. In relieving the pressure from the oxygen pressure chamber preparatory to removing the aged specimens, it is essential that the release be slow and uniform, requiring at least 5 min. so as to avoid possible formation of porosity in the specimen.

NOTE.—For the evaluation of rubber compounds intended for service at elevated temperatures, the above methods may be used with an operating temperature of  $80 \pm 1$  C., employing time intervals as suggested in Paragraph (d), or as otherwise agreed upon. It should be noted that by increasing the aging temperature to 80 C. from 70 C. the rate of oxidation may be expected to be approximately double, and if the rubber compound is of a rapid aging type, or if it is contaminated with such materials as copper or manganese, the rate of oxidation may be catalyzed to such extent as to become violent.

#### Physical Tests of Aged Specimens

9. The tensile strength and ultimate elongation or the stress-strain properties

of the specimens aged for different intervals shall be determined as the intervals terminate in the progress of aging, disregarding the fact that more specimens may still be aging. In determining the physical properties after aging, the final values shall be the average of results from at least two specimens including that one giving the highest value for ultimate tensile strength and any which check that value within 15 per cent. Results of tests of all other specimens shall be discarded. In the event that discarding of aged specimens both from faulty breaks and failure to check does not leave two satisfactory determinations, additional specimens shall be aged and tested until two or more check results from properly broken specimens are obtained. After completion of the tests, the broken aged specimens shall be examined visually and manually and their condition noted.

#### Calculations and Report

10. (a) The results of the aging test shall be expressed as a percentage of the

deterioration in each physical property (tensile strength, ultimate elongation or tensile stress), calculated as follows:

$$\text{Percentage of deterioration} = \frac{O - A}{O} \times 100$$

where:

$O$  = original value, and  
 $A$  = value after aging.

(b) The report shall include the following:

- (1) The results calculated in accordance with Paragraph (a),
- (2) All observed and recorded data on which the calculations are based,
- (3) Type of aging test,
- (4) The aging interval,
- (5) The aging temperature,
- (6) The duration, temperature, and date of vulcanization of the rubber, if known,
- (7) Dates of original and final determinations of physical properties, and
- (8) Dimensions of test specimens.

## *Standard Method of Test for*

### ACCELERATED AGING OF VULCANIZED RUBBER BY THE OVEN METHOD<sup>1</sup>



A.S.T.M. Designation: D 573 - 45

ADOPTED, 1940; REVISED, 1941, 1942, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 573; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. This method is intended for use in estimating the relative resistance of vulcanized rubber to age deterioration. No direct correlation between this accelerated test and natural life of rubber is given or implied. Since the rate at which deterioration proceeds during the normal life of rubber varies widely depending on conditions of exposure to heat, light, and air and on the composition and state of cure of the rubber, this accelerated test is comparative only and must be evaluated against the performance of rubber compounds of which both the natural and accelerated aging characteristics are known.

#### Nature of Test

2. (a) This aging test of rubber consists of subjecting test specimens having previously determined physical properties to controlled deteriorating influences for known periods, after which the physi-

cal properties are again measured and the changes noted. In this method the test involves exposure of specimens to an elevated temperature in air at atmospheric pressure. The test is designated as the air-oven aging test. It does not include exposure to light.

(b) In this aging test, the physical properties used to measure the deterioration of the rubber, in addition to visual and manual inspection, are tensile strength and ultimate elongation or stress-strain relationship. Except as otherwise specified in this method, the determination of these properties before and after aging shall be carried out in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials.<sup>3</sup>

#### General Methods

3. (a) Except as otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> This method formerly comprised the air-oven test procedure in the former Tentative Methods of Test for Accelerated Aging of Vulcanized Rubber (D 428 - 36 T), which was published as tentative from 1935 to 1940, being revised in 1936.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



Materials<sup>3</sup> shall be complied with and are made a part of this method.

(b) In case of conflict between the provisions of this method and those of detailed specifications or methods of test for a particular material, the latter shall take precedence.

#### Apparatus

4. The air oven shall conform to the following requirements:

(a) The interior size shall be as follows or of an equivalent volume:

Interior size of air oven:

Min. ....	12 by 12 by 12 in.
Max. ....	36 by 36 by 48 in.

(b) Provision shall be made for suspending specimens vertically without touching each other or the sides of the aging chamber.

(c) The temperature variation in various parts of the oven shall not exceed 2 C.

(d) The heating medium for the aging chamber shall be air circulated within it at atmospheric pressure.

(e) The source of heat is optional but shall be located in the air supply outside of the aging chamber proper.

(f) A recording thermometer located in the upper central portion of the chamber near the center of the aging specimens shall be provided to record the actual aging temperature.

(g) Automatic temperature control by means of thermostatic regulation shall be used.

(h) The following special precautions shall be taken in order that accurate, uniform heating is obtained in all parts of the aging chamber:

(1) The heated air shall be thoroughly circulated in the oven by means of mechanical agitation. When a motor-driven fan is used, the air must not come in contact with the fan

motor brush discharge because of danger of ozone formation.

(2) Baffles shall be used as required to prevent local overheating and dead spots.

(3) The thermostatic control device shall be so located as to give accurate temperature control of the heating medium. The preferred location is adjacent to the recording thermometer.

(4) An actual check shall be made by means of maximum reading thermometers placed in various parts of the oven to verify the uniformity of the heating.

#### Test Specimens

5. (a) Dumbbell-shaped test specimens, prepared as described in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials<sup>3</sup> shall be considered standard. Their form shall be such that no mechanical, chemical, or heat treatment will be required after aging. If buffing is necessary, it shall be performed prior to the aging cycle.

(b) The dimensions of the test specimens for calculating the physical properties shall be measured prior to starting the aging cycle. Care shall be taken that the material used for marking the gage lines on the specimens is not detrimental to the rubber during aging exposure, causing the specimens to break at the marks prematurely during test. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other.

#### Number of Test Specimens

6. (a) At least three specimens shall be used to determine the average original physical properties of the rubber and also three or more specimens of the same

material for each interval of the aging test.

(b) When minimum requirements are established, one test shall be made for tensile strength and elongation. If the results are below the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to meet the specifications.

#### Tests of Unaged Specimens

7. (a) The stress-strain properties or tensile strength and ultimate elongation of the original unaged specimens shall be determined within 24 hr. of the start of the aging period. Results on specimens which break outside of the straight reduced section or which are found to be imperfect shall be discarded, and retests shall be made.

(b) When rubber compounds are to be tested for the purpose of determining compliance with a specification, it shall be permissible to determine the original properties required in Paragraph (a) simultaneously with the determination of the values after the first aging period even though the elapsed time exceeds 24 hr.

#### Procedure for Accelerated Aging

8. (a) The specimens for aging shall be placed in the oven after it has been preheated to the operating temperature. Simultaneous aging of a mixed group of different compounds shall be avoided if possible. For instance, high-sulfur compounds should not be aged with low-sulfur compounds and those containing antioxidants shall not be aged with those having no age-resistors. Some migration is known to occur.

(b) The operating temperature shall be  $70 \pm 1$  C. (Note).

(c) The aging interval shall start at the time the specimens are placed in the oven and shall continue for a measured time interval. The selection of suitable intervals of aging will depend on the rate of deterioration of the particular material being tested. Intervals frequently used are 2, 4, 7, and 14 days.

(d) The aging intervals used shall be such that the deterioration will not be so great as to prevent determination of the final physical properties. In experimental work, it is desirable to use a range of periods, while for routine tests of known materials fewer intervals may be employed.

(e) At the termination of the aging interval, the specimens shall be removed from the oven, cooled to room temperature on a flat surface and allowed to rest not less than 16 hr. nor more than 48 hr. before determination of the physical properties.

NOTE.—For the evaluation of natural rubber compounds intended for service at elevated temperatures, the above methods may be used with an operating temperature of  $90 \pm 1$  C. and for the evaluation of synthetic rubber compounds for all types of service, the above methods may be used with an operating temperature of  $100 \pm 1$  C., employing in both cases time intervals as suggested in Paragraph (c), or as otherwise agreed upon. It should be noted that, by increasing the aging temperature to 90 C. from 70 C., the rate of oxidation of natural rubber compounds may be expected to be approximately quadrupled, and if the compound is of a rapid aging type, or if it is contaminated with such materials as copper or manganese salts, the rate may be catalyzed to such extent as to become violent. By increasing the aging temperature to 100 C. from 70 C., the rate of oxidation of synthetic rubber compounds may be expected to be greatly increased, depending upon the type of formulations used, especially in respect to the amount of anti-oxidants which they contain.

#### Physical Tests of Aged Specimens

9. The tensile strength and ultimate elongation or the stress-strain properties

of the specimens aged for different intervals shall be determined as the intervals terminate in the progress of aging, disregarding the fact that more specimens may still be aging. In determining the physical properties after aging, the final values shall be the average of results from at least two specimens including that one giving the highest value for ultimate tensile strength and any which check that value within 15 per cent. Results of tests of all other specimens shall be discarded. In the event that discarding of aged specimens both from faulty breaks and failure to check does not leave two satisfactory determinations, additional specimens shall be aged and tested until two or more check results from properly broken specimens are obtained. After completion of the tests, the broken aged specimens shall be examined visually and manually and their condition noted.

#### Calculations and Report

10. (a) The results of the aging test

shall be expressed as a percentage of the deterioration in each physical property (tensile strength, ultimate elongation, or tensile stress), calculated as follows:

$$\text{Percentage of deterioration} = \frac{O - A}{O} \times 100$$

where:

$O$  = original value, and

$A$  = value after aging.

(b) The report shall include the following:

(1) The results calculated in accordance with Paragraph (a),

(2) All observed and recorded data on which the calculations are based,

(3) Type of aging test,

(4) The aging interval,

(5) The aging temperature,

(6) The duration, temperature, and date of vulcanization of the rubber, if known,

(7) Dates of original and final determinations of physical properties, and

(8) Dimensions of test specimens.

# *Standard Method of*

## AIR PRESSURE HEAT TEST OF VULCANIZED RUBBER<sup>1</sup>



A.S.T.M. Designation: D 454 - 41

ADOPTED, 1940; REVISED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 454; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method is intended for use as an accelerated test applicable for estimating the probable life of rubber compounds designed for high-temperature service. It is not suitable for rubber products that have not been specifically designed to withstand elevated temperatures in service. No direct correlation between the test and actual life in service should be implied but when the test is used comparatively between rubber compounds of similar type, the measured changes give valuable indication as to the relative ability of each compound to withstand service conditions involving the same factors as those employed in the test.

### Type of Test

2. (a) The air pressure heat test for rubber consists of exposing test specimens having previously determined physical properties to the deteriorating influence of air at specified elevated

temperature and pressure for known periods, after which the physical properties are again measured and the changes noted.

(b) The physical properties used to measure the deterioration of the rubber, in addition to visual and manual inspection, are tensile stress at a specified elongation, or tensile strength and ultimate elongation, or both. Except as otherwise specified in this method, the determination of these properties before and after each exposure period shall be carried out in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials.<sup>3</sup>

### General Methods

3. (a) Except as may be otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials<sup>3</sup> shall be complied

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1937 to 1940.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

with and are made a part of this method.

(b) In case of conflict between the provisions of this method and those of detailed specifications or methods of test for a particular material, the latter shall take precedence.

#### Apparatus

4. The air-pressure chamber shall consist of a metal vessel designed to maintain an internal atmosphere of air under known pressure with provisions for placing rubber specimens within it and subjecting them to controlled uniform temperature. The equipment shall conform to the following requirements:

(a) The size and shape of the chamber shall be optional but shall be such that the specimens may be suspended therein vertically without undue crowding and without touching each other or the sides of the chamber.

(b) The operating temperature shall be  $126.7 \pm 1$  C. ( $260 \pm 1.8$  F.) determined by measurement at the center of the suspended specimens and shall be automatically controlled by means of thermostatic regulation.

(c) The source and means of heating shall be optional but the uniformity of temperature distribution at various points within the pressure chamber shall be verified by actual check since the test is sensitive to small temperature variations. Local overheating or "dead spots" shall be avoided. Care shall also be taken in the design of the equipment to avoid direct radiation of heat on the samples.

(d) The apparatus and method of heating shall be so designed that the interval required for the chamber to reach the operating temperature at the beginning of a test shall be as short as possible. By proper precautions this lag may be reduced to less than 5 per cent of the usual minimum exposure

periods. Provision shall also be made for rapid closing and opening of the apparatus for introduction or removal of specimens.

(e) The air pressure shall be maintained at  $80 \pm 2.0$  psi. during the exposure periods. Automatic regulation is recommended.

(f) Suitable provision shall be made by separation, filtration, or otherwise for removal of oil, dirt, and moisture from the air entering the pressure chamber. Care shall also be taken to avoid any other introduction of oil or grease into the pressure chamber.

(g) No copper or brass parts shall be exposed to the atmosphere used in the pressure chamber.

(h) The pressure chamber shall be equipped with a reliable safety valve set for release at a pressure of not more than 200 psi.

NOTE: *Caution.*—Adequate safety provisions are important when heating oxidizable organic materials in air under pressure since the rate of oxidation may become very rapid in some cases, particularly if large surface area is exposed.

#### Test Specimens

5. (a) Dumbbell-shaped test specimens, prepared as described in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials,<sup>3</sup> shall be considered standard. Their form shall be such that no mechanical, chemical, or heat treatment will be required after exposure in the pressure chamber. If buffing is necessary, it shall be performed prior to exposure.

(b) The dimensions of the test specimens for calculating the physical properties shall be measured prior to exposure in the pressure chamber. Care shall be taken that the material used for marking the gage lines on the specimens is not detrimental to the rubber during the

heat treatment, causing the specimens to break at the marks prematurely during subsequent test. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other.

#### Number of Tests

6. At least three test specimens shall be used to determine the average original physical properties of each sample and also three or more specimens of the same material for each exposure period of the test.

#### Tests of Original Specimens

7. The tensile stress at a specified elongation or tensile strength and ultimate elongation, or both, of the original unexposed specimens shall be determined within 48 hr. of the start of the heat treatment. Results on specimens which break outside of the straight reduced section, or which are found to be imperfect, shall be discarded and retests shall be made.

#### Procedure

8. (a) The specimens for exposure shall be suspended vertically in the pressure chamber after it has been preheated to the operating temperature. Simultaneous exposure of a mixed group of different compounds shall be avoided, if possible. For instance, high-sulfur compounds should not be exposed with low-sulfur compounds nor those containing antioxidants with those which do not, since some migration is known to occur under the conditions of the test.

(b) The exposure period shall be considered to start when the specimens are placed in the heated chamber which shall be closed immediately and the air pressure applied. This entire operation shall not require longer than 3 min. However, a record shall be kept of the time interval elapsing from the starting

time until the temperature of the chamber reaches 126.7 C. (260 F.). If this interval exceeds 5 per cent of the total exposure time, approximate correction shall be made by adding one-half of the interval to the exposure period. The exposure shall be continuous for the specified time without pressure reduction or opening of the chamber for introduction or removal of specimens.

(c) The selection of suitable periods of exposure will depend on the rate of deterioration of the particular material being tested and should be such that the deterioration will not be so great as to prevent determination of the final physical properties. Intervals frequently used are 3, 5, 8, 12, 20, and 30 hr. In experimental work, it is desirable to employ a range of periods so as to determine the rate of deterioration but, for routine tests of known materials and for purchase acceptance purposes fewer intervals or even a single period may suffice.

(d) At the termination of the exposure period, the air pressure shall be released gradually, this operation requiring at least 5 min. so as to avoid possible formation of porosity in the specimens which shall then be immediately removed from the pressure chamber. The specimens shall be cooled to room temperature on a flat surface and allowed to rest not less than 16 nor more than 48 hr. before determination of the physical properties.

#### Physical Tests of Exposed Specimens

9. The tensile stress at a specified elongation, or tensile strength and ultimate elongation, or both, of the specimens exposed for different periods shall be determined as the intervals terminate, except that it shall be permissible to accumulate specimens for testing together when this does not conflict with

the specified rest period. In determining the final physical properties, the values shall be the average of results from at least two specimens, including that one giving the highest value for ultimate tensile strength and any which check that value within 15 per cent. Results of tests of all other specimens shall be discarded. In the event that discarding of exposed specimens both from faulty breaks and failure to check does not leave two satisfactory determinations, additional specimens shall be exposed and tested until two or more check results from properly broken specimens are obtained. After completion of the tests, the broken specimens shall be examined visually and manually and their condition noted.

#### Calculations and Report

10. (a) The results of the air pressure heat test for each exposure period shall be expressed as a percentage of deterioration in each physical property (tensile

strength, ultimate elongation, or tensile stress), calculated as follows:

$$\text{Percentage of deterioration} = \frac{O - E}{O} \times 100$$

where:

$O$  = original value, and

$E$  = value after exposure.

(b) The report shall include the following:

(1) The results calculated in accordance with Paragraph (a),

(2) All observed and recorded data on which the calculations are based,

(3) Description of the apparatus,

(4) The exposure period,

(5) Statement of condition of exposed specimens,

(6) Dimensions of test specimens,

(7) The duration, temperature, and date of vulcanization of the rubber, if known, and

(8) Dates of original and final determination of physical properties.

# Standard Method of Test for RESISTANCE TO LIGHT CHECKING AND CRACKING OF RUBBER COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 518 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 518; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method of test is intended for use in estimating the comparative ability of soft rubber compounds to withstand the effect of sunlight and weather. It does not apply to the testing of material ordinarily classed as hard rubber.

NOTE.—This method is not suited for use in purchase specifications both because correlation with service life is uncertain and because the results from duplicate specimens tested in different locations do not check. No relation between the results of the test and actual service performance is given or implied. The test is principally of value when used for comparisons between two or more rubber compounds.

## Type of Test

2. This test consists of continuously exposing rubber specimens held under strain in direct natural sunlight and weather for definite periods and observing their deterioration as evidenced by the appearance and growth of minute cracks or crazing on the surfaces. The progressive deterioration may ultimately result in rupture of the specimens.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1938 to 1944.

## Method A. Exposure of Straight Specimens

### Apparatus

3. The apparatus shall consist of the following:

(a) *Mounting Block*.—A rectangular wooden block for supporting the extended specimens. The block shall be 5.5 in. in width and approximately 15 in. in length and shall have a thickness of not less than  $\frac{7}{8}$  in. The block shall have the grain running lengthwise and shall be suitably reinforced on the back to prevent warping. Both of the 15-in. edges of the face carrying the specimens shall be rounded with a  $\frac{1}{8}$ -in. radius. (Fig. 1 shows the details of construction). The block shall be planed smooth and painted with two coats of du Pont clear lacquer or its equivalent.

(b) *Tacks*.—Aluminum tacks, size No. 6, made from Alloy No. 51-S for fastening the specimens to the edges of the wooden block.

(c) *Angle Strips*.—Right-angled aluminum molding strips  $\frac{1}{2}$  in. by  $\frac{7}{8}$  in. of approximately No. 22 gage for shielding the specimens where tacked and bent over the edges of the block. Strips



made from commercial aluminum sheet of alloy No. 2-S are suitable.

### Test Specimens

4. (a) The test specimens shall be rectangular strips 1 in. in width by 6 in. in length cut longitudinally from standard laboratory test slabs having a thickness of 0.075 in. minimum and not to exceed 0.100 in.

(b) Duplicate specimens shall be tested whenever possible.

The aluminum shields shall then be mounted by means of screws on each of the long sides so that the  $\frac{7}{8}$ -in. leg covers the tacked ends of the specimens and the  $\frac{1}{2}$ -in. leg covers the specimen at the bend on to the face of the block.

(b) The extended specimens shall be exposed to the weather and sunlight, preferably on the roof of a building. The block holding the specimens shall be placed at an angle of 45 deg. from the

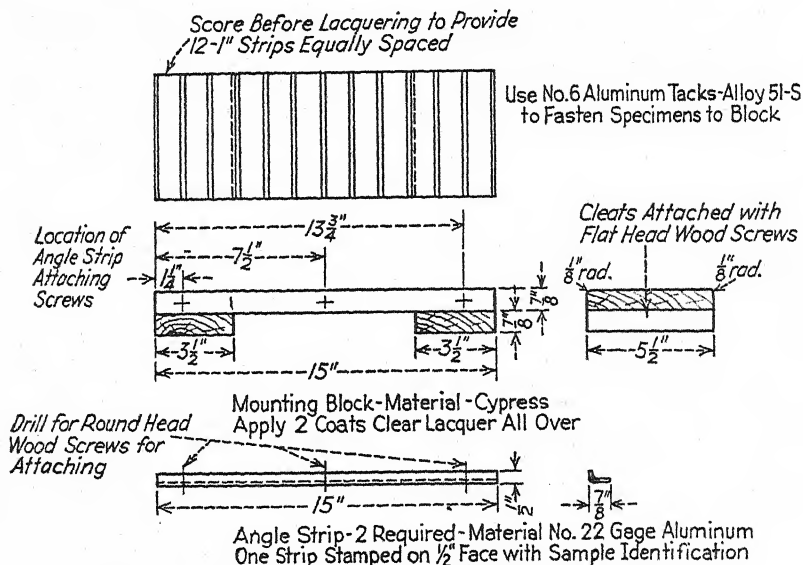


FIG. 1.—Apparatus for Mounting Straight Rubber Specimens, Method A.

### Procedure

5. (a) The test specimens shall be firmly fastened at one end to one long side of the test block spacing them  $\frac{1}{4}$  in. apart and using three aluminum tacks per specimen. The strips shall then be drawn across the face of the block in such a manner as to cause an extension of 20 per cent measured between gage marks which shall be 4 in. apart centered at the middle of each strip. The other end of each specimen shall then be fastened in the same way to the opposite long side of the block.

vertical with the extended specimens facing south.

(c) The date on which the tests were begun shall be recorded and the specimen shall be examined daily thereafter for the effect of sunlight and weather. The time of the appearance of the first surface checking together with the time of the appearance of the first minute surface cracks on each specimen shall be recorded. If desired, the exposure may subsequently be continued for the purpose of observing the rate of growth of the cracks or the development of any characteristic or unusual surface effects.

### Method B. Exposure of Looped Test Specimens

#### Apparatus

6. The apparatus shall consist of the following:

(a) *Clamping Strips*.—Strips made of a medium-soft wood for clamping the specimens. Each strip shall be  $\frac{1}{2}$  in. in thickness, 1 in. in width, and 23 in. in length. Holes shall be drilled through the  $\frac{1}{2}$ -in. thickness of each strip at intervals of  $1\frac{3}{8}$  in. starting  $1\frac{3}{8}$  in. from one end. The holes shall be made using a

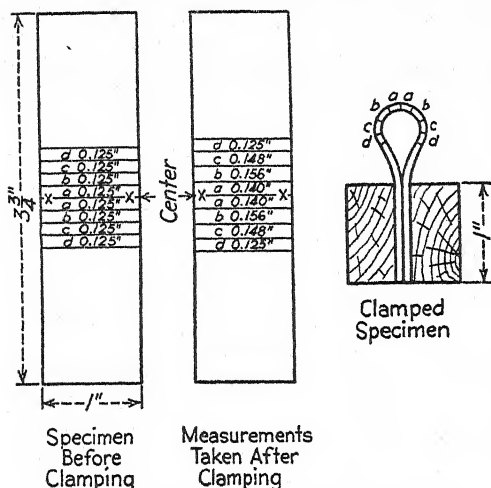


FIG. 2.—Cross-sectional View of Mounted Looped Specimen Showing Elongation at Different Parts, Method B.

No. 22 drill (0.157 in. in diameter) and shall match in paired strips. The strips shall be fastened together using round-head chromium-plated or galvanized iron machine screws (commercial designation: diameter No. 6, thread No. 32, length  $1\frac{1}{2}$  in.) fitted with nuts.

(b) *Base Panel*.—A base panel made of medium-soft wood upon which the clamped specimens are mounted. It shall be 24 in. in length, 8 in. in width, and at least  $\frac{1}{2}$  in. in thickness. Three cross-pieces each 8 in. in length, 1 in. in width, and  $\frac{1}{4}$  in. in thickness shall be fastened to the base. The cross-

pieces shall be mounted  $\frac{3}{4}$  in. from each end of the panel and at the center. Each cross-piece shall be held in place by three 2-in. round-head chromium-plated or galvanized iron machine screws (commercial designation: diameter No. 6, thread No. 32, length 2 in.). These screws shall be fitted with  $\frac{7}{8}$ -in. washers on the underside of the base panel and shall fit into a  $\frac{1}{2}$ -in. counter-sink. The screws shall pass through the panel and through the cross-pieces,  $1\frac{1}{4}$  in. from each end of the cross-pieces and through the center. These screws shall protrude above the surface of the panel to a height of about  $1\frac{3}{8}$  in. and shall be used to fasten the wooden specimen strips securely to the base as described in Section 8 (b).

(c) All wooden panels, strips and cross-pieces shall be painted with two coats of Dulux Suede No. 93503 or duPont Clear Lacquer, or its equivalent.

#### Test Specimens

7. (a) The test specimens shall be rectangular strips 1 in. in width by  $3\frac{3}{4}$  in. in length, cut longitudinally from standard laboratory test slabs having a thickness of 0.075 in. minimum and not to exceed 0.100 in.

(b) Duplicate test specimens shall be mounted whenever possible.

#### Procedure

8. (a) The test specimens shall be looped until their ends meet and these shall then be inserted between the paired wooden strips until they are flush with the underside of the strips. The minimum distance between specimens shall be  $\frac{1}{4}$  in. The wooden strips shall then be clamped together by means of machine screws so that the specimens shall be firmly held in place. As a result of the above procedure, 1 in. of each end of the specimen will be

covered by the wooden strips which will act as a protective shield. The remaining  $1\frac{3}{4}$  in. of the specimen shall form a loop having a varying elongation along its length as illustrated in Fig. 2.

(b) The rack of clamped specimens shall be mounted on the cross-pieces attached to the base panel by passing the protruding machine screws of the panel between the paired wooden strips and fastening with nuts and washers, as illustrated in Fig. 3.

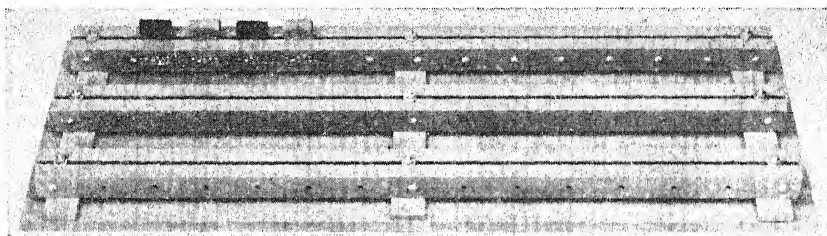


FIG. 3.—Assembled Panel, Showing Method of Mounting Looped Specimens.

(c) The looped specimens shall next be exposed to the weather and sunlight, preferably on the roof of a building. The panel holding the specimens shall be placed at an angle of 45 deg. from the vertical with the specimens facing south.

(d) The specimens shall be examined daily or as often as necessary for the effect of sunlight and weather. The time of appearance of checking as well as the time of appearance of initial

surface cracking shall be recorded for each specimen. If desired the exposure may subsequently be continued for the purpose of observing the rate of growth of the cracks or the development of any characteristic or unusual surface effects.

#### Report

9. For each of the alternative methods, the report shall include the following:

- (a) Statement of method used.
- (b) Description of the specimens

identifying the rubber compounds and giving the duration, temperature, and date of vulcanization if known.

(c) Dates of starting sunlight exposure and first appearance of surface checking and cracking.

(d) Geographical location of the place of exposure.

(e) Description of the appearance of the exposed specimens including, if feasible, a photographic record.

# *Standard Methods of Test for* ADHESION OF VULCANIZED RUBBER (FRICTION TEST)<sup>1</sup>



A.S.T.M. Designation: D 413 - 39

ADOPTED, 1939.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 413; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. (a) These methods cover the procedures for testing the strength of adhesion between plies of fabric bonded with rubber or the adhesion of the rubber layer in articles made from rubber attached to another material. They are applicable only when the adhered surfaces are approximately plane or uniformly circular as in belting, hose, tire carcasses, or rubber-covered sheet metal. When the adhered surfaces contain sharp bends, angles or other gross irregularities which cannot be avoided in preparing test specimens, special methods must be employed for evaluating adhesion. Two general test procedures are given:

*Dead Weight Method* in which the force is applied by means of weights.

*Machine Method* in which the force is applied by means of a tension testing machine.

(b) As covered by these methods an adhesion test shall be understood to con-

sist of applying to a test specimen, under specified conditions, a measured force sufficient to strip from the specimen at a measured rate a layer of moderate thickness with separation at the adhered surfaces. The numerical value of the adhesion may therefore be expressed as (1) average force required to cause separation at a definite rate, or (2) average rate of separation caused by a known or specified force. In the machine method, the rate of separation is fixed and the adhesion value must be expressed in terms of the measured tension. With the dead-weight method, either item 1 or 2 can be used but the latter is preferable and will be here employed.

(c) Except as may be otherwise specified in these methods, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials<sup>3</sup> shall be complied with and are made a part of these methods.

(d) In case of conflict between the provisions of these methods and those of detailed specifications or methods of

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1935 to 1939, being revised in 1936.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

test for a particular material, the latter shall take precedence.

### Test Specimens

2. (a) Test specimens shall conform to one of two types as follows:

*Strip Specimens.*—Plane strips having a width of  $1.00 \pm 0.01$  in., and a minimum length such as to permit separation over sufficient distance to indicate the adhesion value.

*Ring Specimens.*—Closed rings having a width of  $1.00 \pm 0.01$  in. and maximum internal diameter of 4 in.

(b) Test specimens may vary in thickness according to the construction of the article being tested, but the portion from which a ply or layer is being separated shall not be of less thickness than such ply or layer and in no case shall the thickness of the latter exceed  $\frac{1}{4}$  in. If necessary, in order to comply with this requirement, slicing or buffing may be employed.

(c) Curved specimens for test in strip form shall have curvature only in the length dimension of the test specimen.

### Cutting of Specimens

3. (a) If practicable the test specimens shall be cut from the article to be tested in such manner that the adhered parts shall have the same 1-in. width. When necessary, or when so specified, only the layer which is to be separated by application of load may be cut to the 1-in. width but in such case, the portions of that layer remaining outside of the test width shall be removed from the other layers to avoid edge constriction during separation.

(b) Cutting of test specimens shall be done using a sharp tool which will leave clean edges. Ring specimens (as from hose samples) are conveniently prepared by mounting the material on a smooth, close-fitting, slightly tapered wooden mandrel which can be rotated

in a lathe and the 1-in. sections cut with a sharp-pointed wet knife forced gradually through the material.

(c) Rings having internal diameters over 4 in. shall be cut through and opened to form strip specimens.

### DEAD-WEIGHT METHOD

#### Apparatus

4. The apparatus required for the adhesion test by the dead-weight method

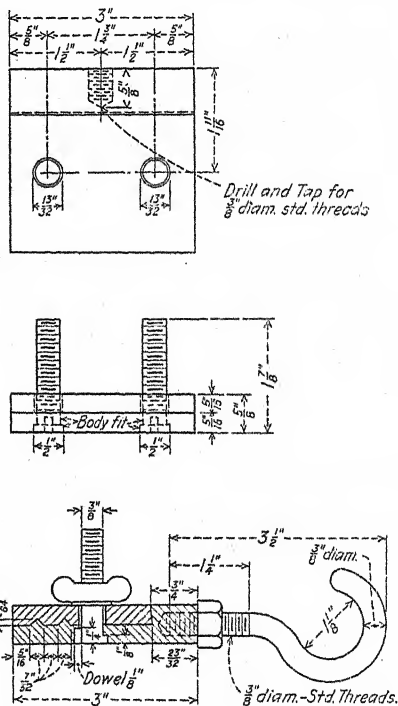


FIG. 1.—Clamp for Adhesion Test.

consists of a supporting frame, testing clamps, mandrels, calibrated weights, and weight carriers. The supporting frame shall be of such design that clamps for strip specimens may be hung on it vertically and that mandrels for ring specimens may be supported on it horizontally. The frame shall have sufficient height to permit weights to be suspended from the test specimens by

means of clamps and to hang freely during the progress of the test. Provision also shall be made to support the mandrels so that they may revolve freely with minimum friction. Suitable apparatus is shown in Figs. 1 and 2.

#### Procedure

5. (a) *Testing Strip Specimens.*—The parts to be tested shall be separated by

men, or the specimen may be held against a vertical plate in such a manner as to keep the specimen in approximately a vertical position during test. A specified or known weight shall be applied by means of a clamp and weight carrier to the layer of which the adhesion is to be determined. The weight of the clamp and carrier shall be included in the total weight causing separation.

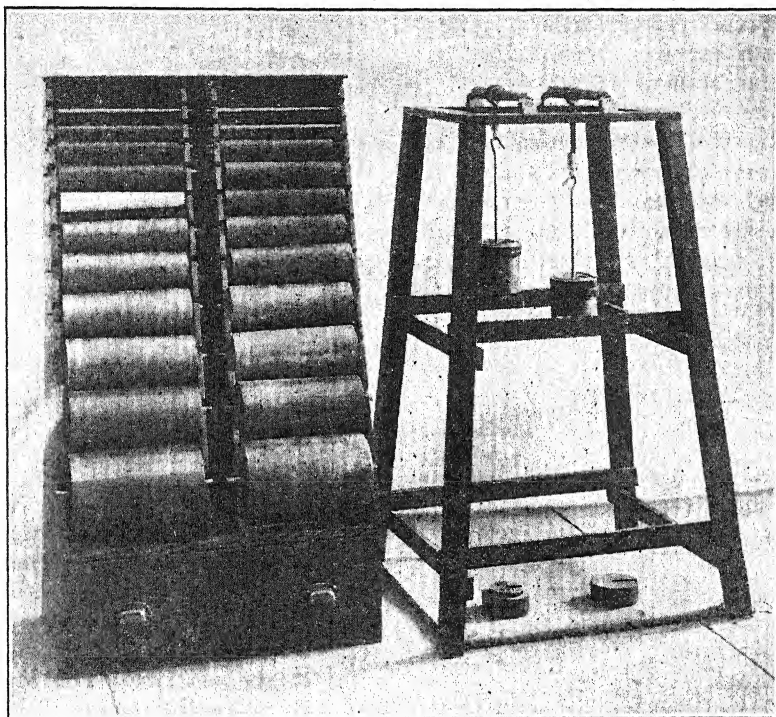


FIG. 2.—Mandrels and Frame for Dead-Weight Adhesion Test.

hand at one end of the strip specimen a sufficient distance to permit attaching the jaws of the testing clamp. The strip shall be suspended from the supporting frame by the separated end, all separate parts of which except the one under test, shall be held in the jaws of the testing clamp. A minimum weight sufficient to maintain the strip specimen in approximately a vertical position shall be attached to the lower end of the test speci-

Suitable provision shall be made for releasing the weight slowly without jerking and in such a manner that the separating layer will be stripped from the specimen approximately at an angle of 180 deg.

(b) *Testing Ring Specimens.*—The ring specimen shall be placed snugly on a mandrel having an outside diameter substantially the same as the internal diameter of the ring. The parts to be tested shall be separated sufficiently by



hand to permit attaching the jaws of the testing clamp. With the mandrel resting on the supporting frame, a known or specified weight shall be applied by means of the clamp and a weight carrier to the layer of which the adhesion is to be tested. The weight of the clamp and carrier shall be included in the total weight causing separation. Suitable provision shall be made for releasing the weight slowly without jerking and in such a manner that the separating layer will be stripped from the specimen approximately at an angle of 90 deg.

(c) *Rate of Separation.*—The rate of separation shall be determined by observing the duration of the test with a stop-watch or a watch having a second hand and by measuring, after the weight has been removed, the length stripped between marks placed at the beginning and end of the test on the other portion than that stripped.

#### MACHINE METHOD

##### Apparatus

6. Adhesion tests by the machine method shall be made on a power-driven apparatus, preferably of the inclination balance or pendulum type, although a spring-balance apparatus may be used. The machine shall fulfill the requirements prescribed in the following Paragraphs (a) to (f):

(a) The applied tension as measured and recorded shall be accurate within plus or minus 1 per cent.

(b) Strip specimens shall be held in the testing machine by grips which clamp firmly and prevent slipping at all times during the test. For ring specimens the grip attached to the recording head of the machine shall be replaced with a freely rotating mandrel having an outside diameter substantially the same as the inside diameter of the ring which is placed on it. The mandrel shall be mounted in such a way that its axis of

rotation will be in the plane of the ply being separated from the ring and that the applied force will be normal to the tangent of the ring circumference at the line of separation.

(c) The rate of travel of the power-actuated grip shall be 2 in. per min. for strip specimens and 1 in. per min. for ring specimens. These rates which provide a separation of 1 in. per min. in both cases shall be uniform throughout the tests.

(d) The machine shall be operated without any device for maintaining maximum load indication. In pendulum type machines, the weight lever shall swing as a free pendulum without engagement of pawls.

(e) The machine shall be autographic giving a chart having the inches of separation as one axis and applied tension as the other axis of coordinates.

(f) The machine shall be of such capacity that the maximum applied tension during the adhesion test shall not exceed 85 per cent nor be less than 15 per cent of the rated capacity.

##### Procedure

7. (a) *Strip Specimens.*—The parts to be tested shall be separated by hand at one end of the test specimen a sufficient distance to permit clamping in the grips of the machine. The separated end of the specimen with all separate parts except the one under test securely gripped shall be attached to the recording head by means of a clamp using care to adjust it symmetrically in order that the tension shall be distributed uniformly. Provision shall be made to maintain the strip during test approximately in the plane of the clamps. This may be done either by attaching the minimum weight required to the free end of the specimen or by holding the specimen against a plate attached to the stationary clamp but, in either case, the

added weight shall be taken into account in determining the load causing separation. That layer of the specimen of which the adhesion is to be tested shall be gripped symmetrically and firmly without twisting in the power-actuated clamp. The autographic mechanism and chart shall be adjusted to zero and the machine started. The separating layer shall be stripped from the specimen approximately at an angle of 180 deg. and the separation continued for a sufficient distance to indicate the adhesion value.

(b) *Ring Specimens*.—With ring specimens that layer of the specimen of which the adhesion is to be tested shall be separated from the specimen by hand sufficiently to permit attaching the power-actuated clamp of the machine. The specimen shall be placed snugly on the test mandrel as described in Section 6 (b). With the mandrel attached to the recording head of the machine and the separated layer gripped symmetrically and firmly without twisting in the power-actuated clamp, the autographic mechanism and chart shall be adjusted to zero and the machine started. The separating layer shall be stripped from the specimen approximately at an angle of 90 deg. to the tangent of the specimen surface and the separation continued for a sufficient distance to indicate the adhesion value. During the test, the mandrel shall rotate freely so as to maintain the line of separation at all times approximately in the same position.

#### PRECAUTIONS, CHECK TESTS, AND REPORT

##### Tearing

8. If, during a test, one of the parts begins to tear instead of separating from the other part of the specimen, the material being torn shall be cut with a knife up to the surface of contact between the two parts and the test started

again. If one of the parts repeatedly tears, instead of separating from the other part, a satisfactory result may be secured in the dead-weight method by reducing the amount of the weight used if this is permitted. In case of repeated tearing when using the machine method, the average load at which tearing occurs shall be taken as the result of the test. In cases of tearing, the results shall be so designated.

##### Effect of Temperature

9. The results of this test are affected by temperature. Elevating the temperature causes a faster rate of stripping under a given load. The temperature during test shall therefore be between 70 and 90 F. (21 and 32 C.) and the actual temperature shall be recorded.

##### Interpretation of Results

10. (a) In the dead-weight method, no stripping is started unless a certain weight is used. Small increments in weight do not progressively cause proportional increases in rate of separation. With increasing weight the rate increases slowly at first and finally very rapidly. Rate of separation must therefore be interpreted carefully. A specimen required to separate not more than 1 in. per min. under 20-lb. weight might be regarded as very inferior if the test result was 5 in. per min. The same specimen might not strip at all under 15-lb. weight and might meet the requirement with 18-lb. weight.

(b) The true adhesion value is not determined in case the specimens repeatedly tear. The test, however, indicates that the strength of adhesion exceeds the strength of the material and that the adhesion value is not less than the result obtained.

(c) Adhesion values may differ between different plies of the same article of plied construction and also at different points along the same ply. The adhe-



sion value of one ply to another is taken as the average result of the test over a reasonable distance of separation. When possible, a distance of separation of at least 4 in. shall be used.

#### Check Tests

11. The adhesion value shall be based on the result of a single test. In case of testing to meet a specified value, one check test shall be made if the average strength as determined by the machine method fails by less than 0.5 lb. to meet the specifications or if the rate of separation determined by the dead-weight method exceeds the specified rate by not more than 10 per cent. The check test result shall be considered final.

#### Expression of Results

12. (a) With the dead-weight method, the value of the adhesion shall be reported as inches of separation per minute per inch of width under a stated weight.

(b) With the machine method, the autographic chart constitutes the report but the value of the adhesion shall be determined by drawing on the chart the best average line between the maximum and minimum load values. The load so indicated, expressed in pounds per inch of width for separation at 1 in. per min., shall be reported as the adhesion value.

#### Report

13. The report shall include the following:

(a) Results of the adhesion test expressed in accordance with Section 12,

(b) All observations and recorded data on which the results are based,

(c) Date of manufacture or vulcanization of rubber, if known,

(d) Date of test,

(e) Statement of method used,

(f) Temperature of test room, and

(g) Dimensions of test specimen.

# *Standard Method of Test for*

## ADHESION OF VULCANIZED RUBBER TO METAL<sup>1</sup>



A.S.T.M. Designation: D 429 - 39

ADOPTED, 1939.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 429; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method covers the procedure for testing the strength of adhesion of rubber to metal in articles where a rubber part is assembled between two parallel metal plates.

(b) The method is designed primarily to apply to specimens prepared in a laboratory under standardized conditions such as may be used to provide data for development and control of rubber compounds and methods of manufacture. With slight modifications as indicated, it may also be used for obtaining comparative adhesion tests of production parts whenever the design permits preparation of suitable test specimens. It is applicable in the case of many automotive parts in which rubber is used for the purpose of damping vibration.

(c) The method does not cover the testing of adhesion of rubber to metal when the part has metal on one side of

the rubber only. For such cases reference should be made to the Standard Methods of Test for Adhesion of Vulcanized Rubber (Friction Test) (A.S.T.M. Designation: D 413) of the American Society for Testing Materials.<sup>3</sup>

(d) Except as otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society of Testing Materials<sup>3</sup> shall be complied with and are made a part of this method.

### Apparatus

2. (a) *Testing Machine*.—A tension testing machine conforming to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials<sup>3</sup> shall be used for measuring the strength of adhesion. The moving head of the machine shall travel at the rate of 1 in. per min. The machine shall be provided with a recording device to give the

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber Like Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1935 to 1939, being revised in 1936.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

total pull in pounds at the conclusion of the test. A machine of not more than 10,000-lb. capacity will be found suitable in most cases.

load during test. Specially molded laboratory specimens shall be attached by means of threaded studs on the metal parts as shown in Fig. 1, but with speci-

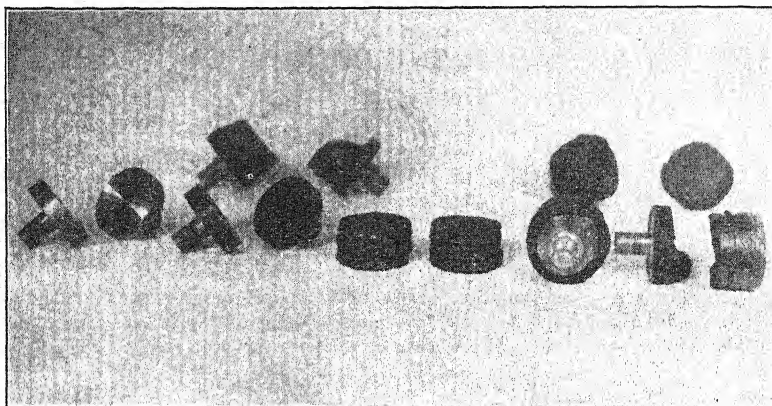


FIG. 1.—Views of Test Specimen Showing Individual Metal Parts and Rubber Cylinders.

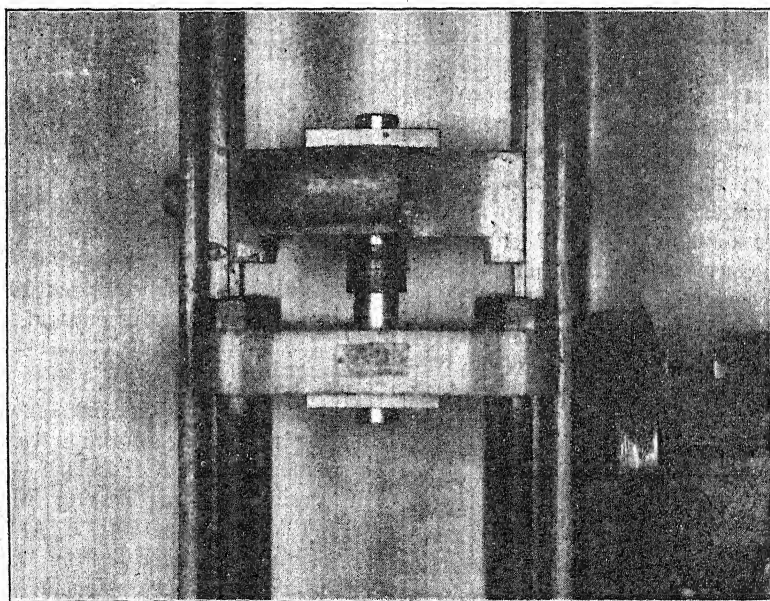


FIG. 2.—Adhesion Test Specimen Attached in Testing Machine.

(b) *Grips*.—The fixtures for holding the specimen in the testing machine will depend on the type of specimen but in all cases shall be provided with ball seats to permit proper centering of the

mens cut from production parts the clamp illustrated in Fig. 4 shall be used.  
**Standard Test Specimens**

3. The standard test specimen shall consist of a cylinder of rubber  $0.500 \pm$

0.005 in. in thickness and  $1.597 \pm 0.001$  in. in diameter having its circular ends attached to the faces of two metal plates each 0.375 in. in thickness and of the same diameter as the rubber cylinder. The metal plates shall have smooth flat faces which shall be parallel in the finished specimen. The cylindrical shape is used to eliminate sharp corners and to give uniform distribution of the pulling force. The specified diameter which provides a cross-sectional area of 2 sq. in. and the  $\frac{1}{2}$ -in. thickness of rubber have been selected

(a) Circular metal parts of standard dimensions shall be machined from rolled bar steel, S.A.E. No. 1020,<sup>4</sup> each having on the back face a round stud  $\frac{1}{2}$  in. in diameter and  $\frac{3}{4}$  in. in length, threaded with 20 threads per inch as shown in Fig. 3. The stud is used for attaching the test piece to the loading fixture in the testing machine. The smoothly machined test faces of the metals shall be brass plated or prepared in accordance with any method for securing adhesion which may be under investigation.

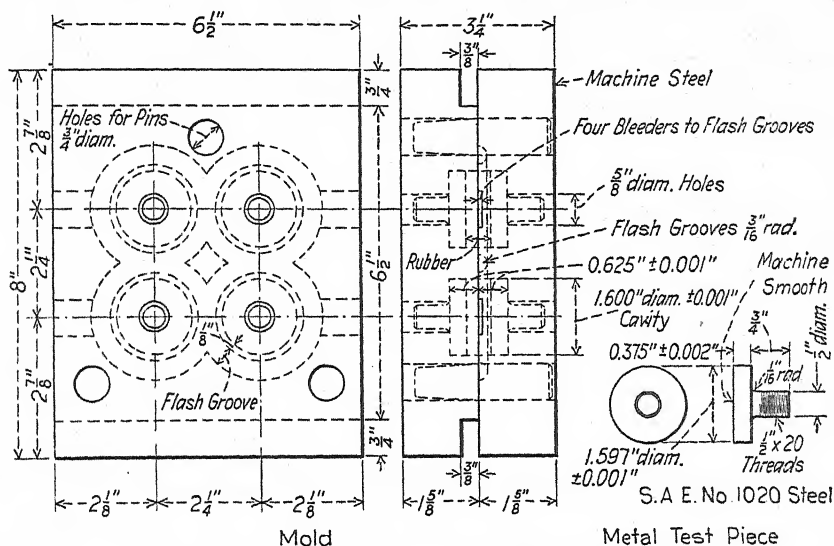


Fig. 3.—Detailed Drawing of Mold for Adhesion Test Pieces.

to provide as large a volume of rubber as advisable without introducing possibility of variations in cure and elongation which would affect the total pull required during test to cause complete separation of the rubber from the metal.

#### Laboratory Preparation of Standard Test Specimens

4. The standard test specimens to be used for development and control purposes shall be prepared in the laboratory as described in the following Paragraphs (a) to (e):

(b) Unvulcanized rubber pieces shall be cut to dimensions of  $1\frac{3}{8}$  in. in diameter and  $\frac{3}{4}$  in. in thickness so as to give maximum pressure of the rubber against the metal surfaces during vulcanization (see Fig. 1). The surfaces to be adhered shall be washed or treated in accordance with the method being investigated.

(c) The metal parts and rubber pieces shall then be assembled for vulcanization in the mold shown in Fig. 3. The method of assembly is illustrated in

<sup>4</sup>1946 S.A.E. Handbook, p. 300.

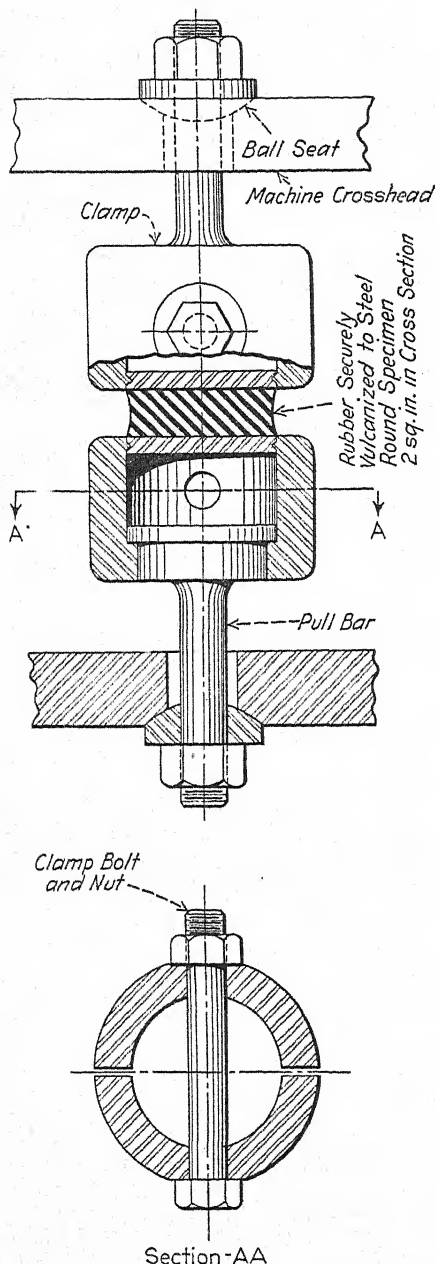


FIG. 4.—Method of Testing Adhesion When Sample Section Is Taken from Production Parts.

Fig. 1. The mold is so constructed that there is obtained after vulcanization approximately 0.0015 in. of rubber over

the edge of the metal to eliminate tearing of the rubber during test from the sharp edge of the metal. Prior to vulcanization great care shall be taken to keep the surfaces to be adhered clean and free from dust, moisture, or other foreign material. The surfaces shall not be touched with the hands.

(d) Vulcanization shall be carried out by heating the mold for a definite time at a controlled temperature in a hydraulic vulcanizing press. The time and temperature of cure shall be chosen according to the rubber compound used. At the conclusion of the cure, care shall be taken in removing the specimens from the mold to avoid subjecting the adhered surfaces to stress before the specimens have cooled.

(e) After vulcanization the specimens shall be kept at room temperature at least 24 hr. before being tested.

#### Test Specimens from Production Parts

5. (a) For comparative tests of adhesion in production parts it is necessary to select a portion of the part such that a specimen consisting of a rubber piece sandwiched between two parallel plates of metal may be cut. The specimen shall be hollow-milled out of the production part and the piece shall be finished either by turning or grinding down to size in such a manner that the surfaces of the rubber and metal shall be free from imperfections which would have any tendency to start a tear in the rubber part during the application of tension while testing.

(b) In routine production testing, it is sometimes necessary to test specimens of different size and shape than those specified as standard. In such cases, the method of preparation shall be similar to that given in Paragraph (a) but the test results secured shall not be compared with those obtained using the standard test specimen.

## Procedure

6. The vulcanized standard test specimen or the specimen cut from a production part shall be mounted in the testing machine, as shown in Fig. 2, using care in centering and adjustment so that the tension shall be uniformly distributed. A steady tension load shall then be applied at the specified machine speed until the rubber either separates from the metal surface or ruptures. The total load at the time of failure shall be recorded. In case of rupture in the rubber, the strength of the adhesion bond is obviously not measured, but is shown to be greater than the strength of the rubber itself. Notation to this effect shall be made on the test report. It is usual, however, when high-grade rubber compounds are used, for the failure to occur at the rubber-metal interface, and the maximum applied pull then truly evaluates the strength of adhesion.

## Check Tests

7. Two tests shall be made and the average value taken if the results check within 100 psi. In the event that the two results fail to check within 100 psi., three additional specimens shall be tested and the average of all five tests shall be reported.

## Calculation

8. The adhesion value shall be expressed in pounds per square inch and

shall be calculated by dividing the tension load causing failure by the original area of the adhered surface which separates. With the standard test specimen, the value is the load at failure divided by two. In case of rupture of the rubber, the value reported is the load at failure divided by the original cross-sectional area of the specimen.

## Report

9. The report shall include the following:

(a) The result calculated in accordance with Section 8,

(b) All observed and recorded data,

(c) Notation of the type of separation and statement of whether the results show true adhesion values or strength of the rubber,

(d) A description of the specimen including statement of method of securing adhesion if known and notation of whether the specimen was molded in the laboratory or prepared from a production part,

(e) Dimensions of the test specimen (comparisons may only be made between specimens of the same size and shape),

(f) Date of manufacture or vulcanization, if known,

(g) Time and temperature of vulcanization, if known,

(h) Temperature of test room, and

(i) Date of test.

# *Standard Methods of Test for*

## ABRASION RESISTANCE OF RUBBER COMPOUNDS<sup>1</sup>



**A.S.T.M. Designation: D 394-46**

ADOPTED, 1940; REVISED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 394; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These methods are designed to measure the abrasion resistance of soft vulcanized rubber compounds, such as gum stocks, boot and shoe stocks, tread stocks, etc. No relation between this test and service performance is given or implied. The significance to be attached to the results can only be determined by each laboratory for its particular problem. It shall not be used for purchase specifications because of the difficulty of reproducing comparison standard compounds in separate laboratories. The value of having the test in standardized form for intercomparisons and for the interpretation of results is, however, unquestioned.

(b) These methods cover three test procedures using the following different types of apparatus:

Method A. E. I. du Pont de Nemours and Co. Abrader.

Method B. National Bureau of Standards Abrader.

Method C. United States Rubber Co. Abrader.

### Type of Materials Tested

2. Any vulcanized rubber compound, whether prepared experimentally in the laboratory, taken from process during manufacture, or cut from a finished article of commerce (providing a section of sufficient size is available), may be tested. It is assumed that the bulk of the testing will be on compounds especially designed to withstand abrasion, and that the test will be carried out with a comparison standard of the same general characteristics.

### Comparison Standards

3. In order to cover the large variety of compounds that can be tested under this method, the three standard compounds prescribed in Table I are recommended as comparison standards. The sample shall be compared with that comparison standard having physical properties (stress-strain values and hardness) most nearly matching the sample compound.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1934 to 1940, being revised in 1937 and 1940.



Preparation of Comparison Standard<sup>3</sup>

4. Having determined which of the three comparison standards is to be used, the following procedure shall be followed:

(a) *Weighing Ingredients.*—The weighing of all ingredients shall be accurate to within 0.25 per cent of the weight specified. The final weight of the mixed batch and the sum of the weights of each

passing through the rolls and a uniform initial temperature, it is certain the mixing process can be made quite uniform.

(d) *Mill Opening.*—It is essential that the relation between batch volume and mill opening shall be regulated to develop an active bank. Since it is neither practical nor economical to standardize the size of the batch, it is recommended that mill openings be adjusted to the

TABLE I.—COMPARISON STANDARDS.

	Parts by Weight <sup>a</sup> Comparison Standards		
	A	B	C
First latex pale crepe.....	50	.....	.....
No. 1 smoke sheet.....	50	100	100
Zinc oxide.....	5	20	5
Channel carbon black.....	.....	30	45
Stearic acid, c. p.....	1	2	4
Medium pine tar.....	.....	.....	4
Mercapto-benzo-thiazole.....	1	.....	1
Diortho-tolyl-guanidine.....	.....	1.25	.....
Phenyl-beta-naphthylamine.....	1	1	1
Sulfur.....	3	3.5	3
Cure.....	60 min. at 259 F. (126 C.) (20 lb. steam)	60 min. at 287 F. (142 C.) (40 lb. steam)	70 min. at 259 F. (126 C.) (20 lb. steam)

<sup>a</sup> It is recognized that the compounding materials shown above may vary and should be completely defined. The attention of Committee D-11 on Rubber Products has been directed toward this problem in the hope of arriving at a satisfactory solution. After extensive cooperative test investigation, it has been established that, with test specimens for Method A prepared from comparison standard compound B in a central laboratory, the volume loss per horsepower hour can be maintained at 200 cu.cm.  $\pm$  5 per cent when determined by the standard procedure using standardized abrasive paper. The laboratories of V. L. Smithers, Inc., 2706 First Central Tower, Akron, Ohio, are prepared to furnish such certified test specimens together with abrasive disks and instructions for calibrating machines and methods to this standard.

ingredient shall not differ by an amount exceeding 0.6 per cent for a compounded stock or 0.3 per cent for pure gum or master-batched stocks.

(b) *Mill Data.*—The rolls of the laboratory mill shall be 6 in. in outside diameter by 12 in. in length with 10½ in. between guides. The speed of the slow roll shall be 24 rpm. and the gear ratio 1.4 to 1.

(c) *Mill Temperature.*—The temperature of the water entering the rolls shall be maintained at 158 F. (70 C.) and the initial temperature of the rolls shall be 158 F. (70 C.). With sufficient water

volume of batches as shown in the following table:

Volume of Batch, cu. cm.	Distance Between Rolls, in.
1200.....	0.170
1100.....	0.160
1000.....	0.145
900.....	0.130
800.....	0.120
700.....	0.110
600.....	0.100
500.....	0.085
400.....	0.070
300.....	0.055

(e) *Mixing Procedure.*—During breakdown, the mill opening shall be 0.055 in. until the rubber runs smooth on the roll, and then the opening shall be made to correspond to the volume of the batch indicated in the table in Paragraph (d). The order of adding ingredients to the broken-down rubber shall be as follows:

<sup>3</sup> This procedure has been adapted from the "Tentative Standard Laboratory Procedure for the Preparation and Physical Testing of Rubber Samples," prepared by the Physical Testing Committee, Rubber Division, Am. Chemical Soc., *The Rubber Age* (New York), January 28, 1930.



## Accelerators and antioxidants

Black

Fillers

Softeners

Sulfur

The ingredients shall be incorporated as rapidly as possible and when all are in the rubber the batch shall be cut six times two-thirds of the distance across the roll, held until the bank just disappears alternating from one side to the other. Then the batch shall be cut across and rolled six times inserting the roll endwise five times and the last time it shall be inserted lengthwise. As soon as the bank is well balanced, the mill opening shall be set to give a sheet slightly greater in thickness than the test specimen after cooling, and the batch shall then be cut from the rolls.

(f) *Storage of Mixed Batch.*—The batch shall be laid upon a suitable surface such as zinc, holland, or talced zinc until cool, after which it shall be stored 18 to 24 hr. on galvanized wire screen (6 mesh is convenient) in subdued light with free circulation of air having a relative humidity of 45 per cent at 82 F. (27.6 C.).

NOTE.—Variation of 0 to 100 per cent relative humidity may cause a difference of 25 per cent in physical properties.

(g) *Preparation for Curing.*—The uncured stock shall be cut with a die having dimensions slightly less than those of the mold. Care shall be taken to have the stock so placed in the mold that the direction of the grain will be parallel to the direction of abrasion when the vulcanized specimen is mounted in the abrader.

(h) *Mold Dimensions.*—Mold dimensions shall be governed by the test specimen needed for the test used.

(i) *Cleanliness of Molds.*—No preparation of any kind shall be used on molds to prevent sticking. The molds

shall be kept clean, and shall be cleaned as soon as the cured stock tends to stick. Three cleaning materials are suggested: (1) ground emery and water, (2) buffer cloth, and (3) whiting paste.

(j) *Maintenance of Curing Temperature.*—The specified curing temperature shall be interpreted as the inside temperature of the mold as determined by a thermocouple or a mercury thermometer in a mercury well in the mold. A similar steel block with a mercury well may be substituted for the mold.<sup>4</sup> To avoid cool spots in platens due to condensation, presses with bored steel platens are recommended. If chamber type platens are used, good drainage should be insured by placing the steam outlet slightly below the bottom of the steam chamber.

(k) *Mold Temperature and Protection.*—The mold shall be brought to the specified curing temperature in a closed press for a period of not less than 20 min. before inserting the uncured stocks. While curing, the mold shall be protected from drafts, by the use of some sort of shields or wooden ells. Protection often causes a rise of 1.8 F. (1 C.) in mold temperature.

(l) *Timing of Cure.*—The time of cure shall start at the time the ram pressure reaches its maximum and end at the release of the ram pressure.

(m) *Cooling Cured Slabs.*—The slabs shall be removed from the mold immediately after conclusion of the cure and plunged into water for at least 10 min. to cool. The water shall be changed frequently to prevent contamination and heating. When removed from the water the slabs shall be wiped dry and placed upon liner or screen for storage in subdued light.

(n) *Conditioning of Specimens.*—The

<sup>4</sup> See Report of Physical Testing Committee, Rubber Division, Am. Chemical Soc., *Industrial and Engineering Chemistry*, Vol. 17, No. 5, p. 535, May, 1925.

test specimens shall be conditioned for 24 hr. in air having a relative humidity of 45 per cent at 82 F. (27.6 C.) before testing at the same temperature.

(o) *Preparation for Test.*—That face of the test specimen which is to be abraded shall be lightly buffed before test, except in those cases where otherwise specified.

(b) If stock is taken from production, the sample shall be prepared as described in Section 4 (f) to (p), inclusive, for the preparation of the comparison standard.

(c) If an article of commerce is being tested, a piece somewhat larger than the test specimen required shall be cut from the sample and subsequently trimmed and buffed to size.

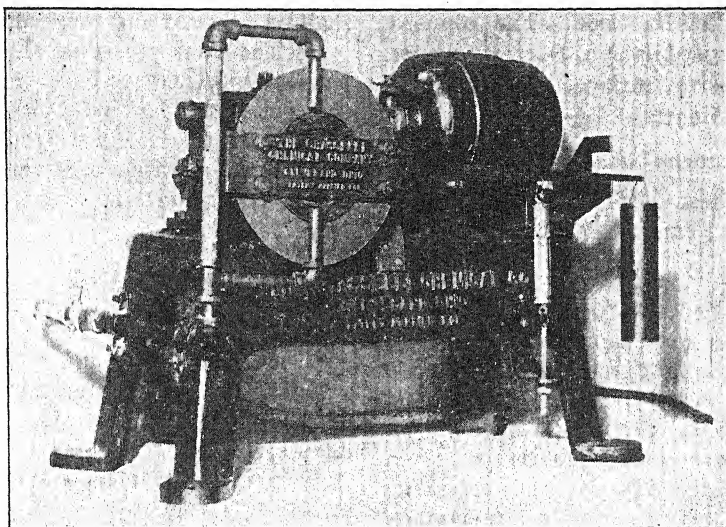


FIG. 1.—E. I. du Pont de Nemours and Co. Abrader.

(p) *Maintenance of Standards.*—It is recommended that sufficient comparison standard compound be prepared at one time to last approximately one month. Subsequent batches of comparison standard shall be tested for stress-strain properties and hardness and shall not vary more than 10 per cent from the original standard adopted. Its abrasion resistance shall be within 5 per cent of the batch of comparison standard being used at that time.

#### Preparation of Sample

5. (a) If a laboratory comparison is being made, the sample shall be prepared by the same procedure as described in Section 4 for the preparation of the comparison standard.

#### METHOD A. E. I. DU PONT DE NEMOURS AND CO. ABRADER<sup>5</sup>

##### Apparatus

6. The essential features of the apparatus, illustrated in Fig. 1, are as follows: A disk which carries an abrasive surface is mounted on a hollow shaft and rotates counter-clockwise in a vertical plane at a speed of 37 r.p.m. Two test specimens are mounted on the inside of a bar, one placed at each end so that their centers are a distance of 5 in. apart. This bar is attached permanently to a rod (axis) which extends through the hollow shaft carrying the abrasion disk. A weight attached to

<sup>5</sup> Ira Williams, "Measurement of Abrasion Resistance of Rubber," *Industrial and Engineering Chemistry*, Vol. 19, No. 6, p. 674, June, 1927.

the end of this rod by means of a cable and acting over a pulley holds the test specimens against the abrasive. A lever arm is attached to the end of the bar which holds the test specimens. The other end of this lever arm carries a bucket, the weight of which is adjusted with shot, just enough to prevent rotation. A vernier spring balance is attached to the lever arm for final adjustment of the load. The abrasive generally used is No. 0 emery paper. The abrasive surface is cleaned by means of air jets.

#### Test Specimens

7. The test specimens shall be 2 cm. square by 1 cm. in thickness.

#### Procedure

8. (a) Two test specimens shall be inserted in the clamps of the balance lever and the clamps then tightened uniformly, care being taken to avoid distorting the surface to be abraded. The balance lever shall be placed in position with its axis rod extending through the hollow shaft which supports the abrasive disk.

The 3.62-kg. weight shall be connected to the end of the axis rod, leveling the cable over the grooved pulley, thus holding the test specimens evenly and firmly against the abrasive disk. The balance weight (bucket) shall be attached to the end of the lever arm and the spring balance connected to the lever arm (for most tests the total weight of the bucket and shot will be 500 g.).

The surface of the abrasive disk shall first be cleaned by applying 20-lb. air pressure to them through the jets. The air shall be filtered to remove oil, water and dirt. Any evidence of these appearing on the abrasive disk shall be cause for repeating the test. The machine shall be run until the test specimens are seated evenly; the specimens

then removed and weighed. The test specimens shall then be replaced in exactly the same position as before and run for 20 min., adjusting the load applied by means of the spring balance to offset rotation of the balance lever. The lever arm will fluctuate slightly between the stops provided on the machine, but should not touch the stops. At the completion of the test, the specimens shall be removed and weighed.

(b) The same procedure shall be followed inserting two test specimens of the comparison standard in the clamps.

(c) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 15.

#### METHOD B. NATIONAL BUREAU OF STANDARDS ABRADER<sup>6</sup>

##### Apparatus

9. The essential features of the apparatus, illustrated in Fig. 2, are as follows: The rubber-coated metal drum, *A*, is 6 in. in diameter and is rotated at  $45 \pm 5$  rpm. by an electric motor with a reducing mechanism, the revolutions of the drum being indicated by a counter attached to one end of the shaft. The three arms, *B*, are each pivoted at one end, and each having a weight suspended at the other end, such that a downward force of 5 lb. is exerted directly on the test specimen on the underside of the arm, *H*. The three dial gages, *C*, graduated in thousandths of an inch, are fastened to a bridge so that each contacts with the corresponding arm, *B*, at a point directly over the test specimen. This bridge is hinged at one end to allow the arms to be swung back for the placing of test specimens on them. The compressed air line, *D*, is provided for keeping the abrasive surface clean.

<sup>6</sup> P. A. Sigler and W. L. Holt, "A Simple Abrasion Test Machine for Rubber," *India Rubber World*, Vol. 82, No. 5, p. 63, August, 1930.

Arm stops, *E*, and an abrasive, *F*, consisting of No. 2½ garnet paper or cloth 6 in. in width, are held on the rubber-covered drum by four rubber bands. The ends of the abrasive sheet are cut at an angle of about 80 deg. and when in place have a clearance of about  $\frac{1}{16}$  in. Test specimens, *G*, 1 in. square and approximately  $\frac{1}{4}$  in. in thickness, are cemented to small fiber disks which in turn are fastened to the weighted arms.

#### Test Specimens

10. The test specimens shall be 1 in. square by  $\frac{1}{4}$  in. in thickness.

average of these readings gives the abrasion resistance in terms of "revolutions per 0.1 in. wear."

(b) For purposes of conforming to this method, the weight of the test specimens before and after abrading may be measured and the average volume loss calculated.

(c) The same procedure shall be followed using three test specimens of the comparison standard on the arms.

(d) From the loss in weight, the specific gravity of the materials and the number of revolutions recorded, the volume loss

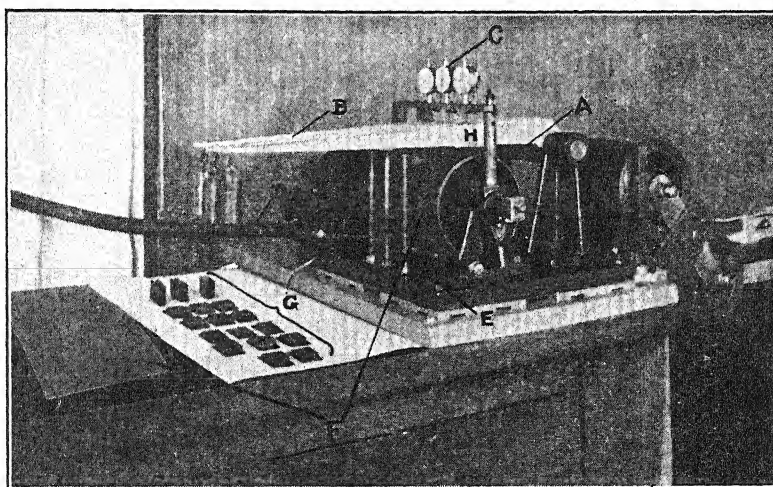


FIG. 2.—National Bureau of Standards Abrader.

#### Procedure

11. (a) In making a test, three specimens shall be mounted, one on each arm and shall first be allowed to wear until the surface conforms to the shape of the drum. The machine shall then be stopped, the gage bridge locked in place, and all gages and the counter set at zero. The machine shall then be run until about 0.1 in. has been abraded from the specimens, and the machine again stopped, and the counter and gage readings recorded. The readings shall always be taken with the drum in the same position. For routine work, the

per 100 revolutions and the abrasion resistance shall be calculated as indicated in Section 15.

#### METHOD C. UNITED STATES RUBBER CO. ABRADER<sup>7</sup>

##### Apparatus

12. The essential features of the apparatus, illustrated in Fig. 3, are as follows: A rotating wheel 3 in. in diameter with the outer rim 1 in. in width

<sup>7</sup> Described in catalogue of Scott Testers, Inc., Providence, R. I.

and covered with an abrasive cloth (metallic cloth, 60 grit). A specimen carrier moves the test specimen held in a suitable grip against this wheel, the wheel rotating against the direction of the movement of the table. The rotation of the wheel is reversed at the time the movement of the specimen table is reversed. Mechanical means are pro-

is equipped with a counter which registers the number of cycles or applications of the abrasive wheel to each specimen.

#### Test Specimens

13. The test specimens shall be slabs 2 by 3 by  $\frac{3}{8}$  in. or 2 by 5 by  $\frac{3}{8}$  in. to fit the standard machine.

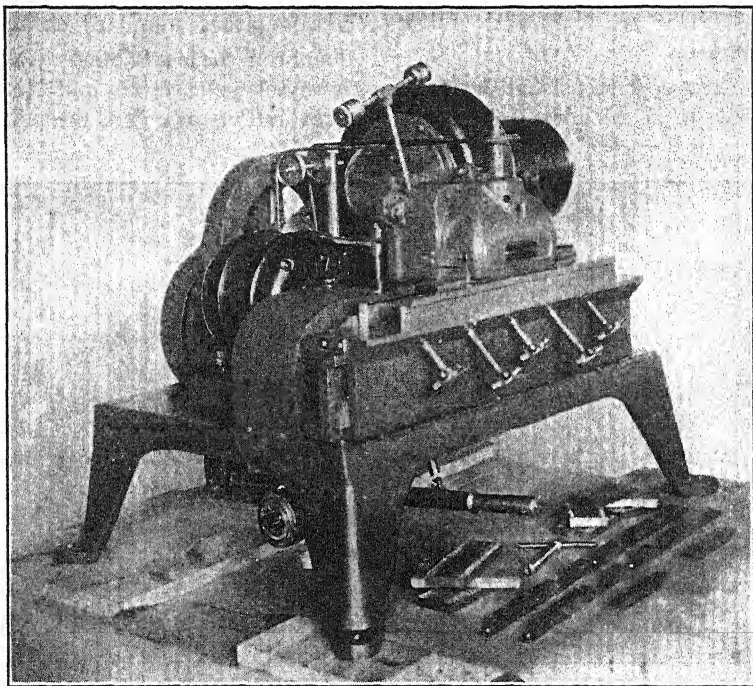


FIG. 3.—United States Rubber Co. Abrader.

vided to raise the wheel from the work as it passes from specimen to specimen. An exhaust blower is provided to remove all dust from the wheel and specimens, and a small revolving brush to clean the wheel thoroughly as it is raised between the grinding of each specimen. This will insure against the carrying of particles from one specimen to the next. A balance arm is provided for the purpose of regulating the pressure of the wheel on the specimens. The machine

#### Procedure

14. (a) After the specimens to be tested have been weighed, they shall be placed in the machine and firmly fastened in place, care being taken that the faces to be abraded are all in the same plane and exactly flush with the restraining edges of the traveling table. A fresh piece of abrasive cloth shall be used for each test.

(b) The machine should be at the end of a cycle so that when started,

each specimen will receive the same number of applications of the abrasive wheel provided that at the conclusion of the test the machine is again stopped at the end of a cycle. The machine is geared to make nine cycles per minute and the length of the test to be made will necessarily depend upon the type of stock being tested. The test shall be run long enough to remove sufficient quantity of the specimen by abrasion to reduce the percentage of error to a minimum. A general rule which should apply requires that the test run until the specimen has been abraded to such a depth that there is still 0.15 in. (0.039 cm.) of material at the point of the deepest cut. The test shall be stopped at a convenient time when abrasion has proceeded to practically this point, at an even number of cycles to facilitate the calculation of results to a common basis for comparison. The test specimens shall then be removed and weighed after brushing them off to remove any loose particles which might remain adhering to the specimens.

(c) From the loss in weight, the volume loss and abrasion resistance shall be calculated in accordance with Section 15.

#### CALCULATIONS

##### Calculations

15. (a) In each of the three alternative methods of test described in Sections 6 to 14 the loss in weight of the test specimen shall be accurately measured to within plus or minus 1 mg., unless otherwise specified.

(b) The specific gravity of the stock shall be determined to the nearest 0.01. The volume loss shall be calculated by dividing the loss in weight by the specific gravity.

(c) The abrasion resistance shall be calculated by dividing the volume loss of the standard by the volume loss of the sample and multiplying by 100.

(d) The results shall be expressed as a percentage of the comparison standard specified.

NOTE: *Example.*—110 per cent of A.S.T.M. Comparison Standard A.



# *Standard Method of Test for*

## TEAR RESISTANCE OF VULCANIZED RUBBER<sup>1</sup>



A.S.T.M. Designation: D 624 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 624; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method describes a procedure which is widely used for determining tear resistance of the usual grades of soft vulcanized rubber such as tire tread, carcass, and inner tube compounds, or those used in most mechanical rubber goods. It does not apply to the testing of material ordinarily classed as hard rubber. Since tear resistance is affected to a large degree by mechanical fibering of the rubber under stress as well as by stress distribution, speed of stretching, and size of specimen, the results obtained in a tear resistance test can only be regarded as a measure of the resistance under the conditions of that particular test and not necessarily as having any relation to service value. The method is useful therefore only for laboratory comparisons and is not applicable for service evaluations, except when supplemented by additional tests, nor for use in purchase specifications. The significance to be attached to the

results can only be determined by each laboratory for its particular problem.

### Apparatus

2. (a) Tear resistance shall be measured on a power-driven apparatus, preferably of the inclination-balance or pendulum type. A spring-balance type of apparatus may be used if provided with a device that will indicate the actual maximum load at which rupture took place and if provision is made to prevent recoil of the spring. The machine shall conform to the following requirements:

(b) The applied load as indicated by a dial or scale shall be accurate within plus or minus 1 per cent.

(c) The indicator shall remain at the point of maximum load after rupture of the test specimen.

(d) The grips which hold the specimen in the testing machine shall have jaws of the narrow cam or pincer type.

(e) The rate of travel of the power actuated grip shall be 20 in. per min. and shall be uniform at all times.

(f) The testing machine shall be calibrated in accordance with the provisions of Section 3.

(g) The testing machine may be

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1944.

equipped with a dynamometer head of the compensating type for convenience in eliminating calculations.

### Calibration of Apparatus

3. The testing machine shall be calibrated by dead-weights applied in an ascending order while the machine is otherwise arranged in an entirely similar manner to that used when testing material. In each case, when additional calibrating weight is added the weight lever of the machine shall be moved to zero position and allowed to swing slowly upwards to the point where it will go no further. The dial of the machine should then clearly indicate the amount of the weight applied.

### Preparation of Sample

4. Except as may be otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials<sup>3</sup> shall be complied with and are made a part of this method.

### Die for Test Specimens

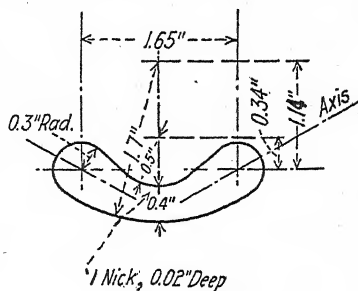
5. The specimens for tear resistance tests shall be stamped out with a steel die. The die shall be kept sharp and free from nicks to avoid leaving ragged edges on the rubber. Cutting may be facilitated by wetting the rubber surface and the cutting edges of the die. The rubber shall be rested on a smooth, slightly yielding surface that will not injure the blade. Lightweight cardboard or a piece of leather belting is suitable. Care shall be taken that the cut edges are perpendicular to the other surfaces of the specimen and have a minimum of concavity.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

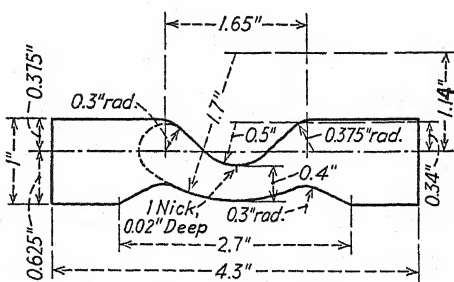
NOTE.—It is a well-known fact that vulcanized rubber exhibits a grain effect which influences its physical properties. In the case of heavily loaded compounds, the effect may produce a pronounced difference in physical properties. Usual practice is to test with the grain running the long way of the specimen, and it is assumed that, unless otherwise specified, all test specimens are to be prepared in this manner. Where grain effects are significant and are to be evaluated, two sets of test specimens should be cut from the sample at right angles to each other.

### Test Specimens

6. (a) The test specimens shall conform in shape to die A or B as shown in Fig. 1, and shall not vary by more than



Die A Specimen.



Die B Specimen.

FIG. 1.—Specimens for Tear Test.

0.5 per cent from the dimensions shown. The thickness shall not fall outside the limits 0.07 to 0.11 in. In general, die B may be expected to give results approximately 10 per cent lower than those obtained when using die A.



(b) The thickness of the specimen shall be measured with a micrometer graduated to 0.001 in., having a presser foot  $0.25 \pm 0.01$  in. in diameter exerting a total force of  $3.0 \pm 0.1$  oz. The load shall be applied by means of a weight.

(c) A single slit or nick shall be cut with extreme care and accuracy across the edge of the specimen at the center of the inner concave edge. In cutting the slit, the specimen shall be held in a suitable device provided with guides for the knife holder to control the depth and centering of the slit. The cutting shall be done by a safety razor blade clamped in a holder with the extension of the blade beyond the surface of the holder regulated by a depth gage block to produce a slit 0.02 in. in depth. In cutting the slit, the specimen shall first be lubricated with soap solution and the knife holder shall then be passed back and forth through the guides of the specimen holder until the slit is cut to the full depth of the razor blade extension.

#### Number of Test Specimens

7. Three specimens per sample shall be tested for tear resistance. In case the tear resistance fails to check within 20 per cent of the highest value obtained, additional specimens shall be tested until two or more such check results are obtained. The final value shall be the average of the check tests, discarding the determinations on specimens that are outside the above limit.

#### Procedure

8. The specimen for test shall be clamped in the jaws of the testing machine, care being taken that the jaws

grip the specimen so that the axes shown in Fig. 1 are in line with the direction of the application of the load. Increased stretch is thus given to the inner curved edge. The "bite" of the jaws shall be at the center of the enlarged ends of the specimen. The load shall then be applied with a lower jaw speed of 20 in. per min. After rupture of the specimen, the breaking load in pounds shall be noted from the dial or scale and recorded together with the average thickness of the specimen.

#### Calculation

9. The resistance to tear shall be calculated from the maximum load registered by the testing machine and the average thickness of the specimen, and shall be expressed as the pull in pounds required to tear a specimen 1 in. in thickness.

NOTE: *Example.*—If a load of 36 lb. is necessary to tear a specimen 0.082 in. in thickness, the tear resistance would be  $= \frac{36.0}{0.082} = 439$  lb. per in.

#### Report

10. The report shall include the following:

(1) Results calculated in accordance with Section 9,

(2) All observed and recorded data on which the calculations are based,

(3) Date of test and date of vulcanization of rubber, if known,

(4) Test room temperature,

(5) Type of testing machine used, and

(6) Designation of die used in preparing the test specimens.

# Standard Method of Test for

## HARDNESS OF RUBBER<sup>1</sup>



A.S.T.M. Designation: D 314 - 39

ADOPTED, 1934; REVISED, 1939.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 314; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method of test covers the procedure for determining the hardness of the more common grade of rubber such as tire treads, most automotive mechanical rubber parts, etc., but not extremely hard or extremely soft rubbers which fall beyond the range of the instrument specified.

(b) For the purpose of this test, the hardness of rubber shall be considered as that property by virtue of which the surface and adjoining layers resist indentation when subjected to a definite pressure by an indentation element of standard size, the contact surface of which conforms in shape to a portion of a true spherical surface.

### A.S.T.M. Hardness Number

2. The A.S.T.M. Hardness Number is the indentation in thousandths of an inch determined under definitely prescribed conditions.

### Apparatus

3. The apparatus consists of a table

for supporting the test specimen, an indenter acting under a dead-weight load, an annular presser foot exerting a definite force on the surface of the specimen surrounding the indentation area, and a suitable device for indicating the depth of indentation.

(a) *Table*.—The table used to support the test specimen shall possess a flat horizontal surface of minimum linear dimension of 2 in.

(b) *Indenter*.—The indenter shall consist of a rigid vertical shaft, the lower end of which is finished to a true hemisphere  $0.0938 \pm 0.0005$  in. in diameter. The maximum diameter of that portion of the shaft which passes through the presser foot shall not exceed that of the indenter point. The point shall be made of a highly-polished, noncorrosive, hard metal properly treated to resist wear due to continuous use (a plated point is not satisfactory). The indenter shall be actuated by a freely acting dead-weight load of 3 lb.  $\pm$  0.25 oz. Less than 0.25 oz. shall be required to overcome friction and to produce perceptible motion of the penetrator when counter-balanced. The indenter shall act through the opening in the center of the presser foot.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.  
<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1929 to 1934, being revised in 1931.

(c) *Presser Foot*.—The presser foot shall be 0.625 in.  $\pm$  0.0005 in. in diameter and shall have a hole 0.109  $\pm$  0.005 in. in diameter located at the center. It shall be sufficiently thick to insure rigidity and the edges shall be rounded to a radius of  $\frac{1}{16}$  in. The inner surface of the presser foot shall be parallel to and concentric with the surface of the shaft of the indenter. The contact surface of the presser foot shall be at right angles to the inner surface, and shall be parallel to the table which supports the test specimen. The presser foot shall be subjected to a freely acting load of 5 lb.  $\pm$  0.75 oz.

(d) *Gage*.—Vertical movement of the indenter shall actuate a suitable dial gage graduated to show indentation in 0.001 in. The accuracy over the whole range shall be within plus or minus 0.0005-in. indentation. The gage shall be mounted so as to be rigid with the presser foot. The indentation measured during the test will therefore be the distance between the contact surface of the presser foot and the tip of the ball point of the indenter.

#### Test Specimens

4. (a) When the instrument is to be used as a reference standard for the calibration of other types of hardness instruments or in arbitration tests, uniform test specimens at least  $\frac{1}{2}$  in. in thickness with parallel sides (slab form) shall be used. The minimum lateral dimension of the specimen shall be 2 in.

(b) In routine testing, specimens may be used which have dimensions other than those required in the procedure for standard tests (Paragraph (a)). In all cases the specimens tested shall for comparison be of essentially the same thickness and size.

#### Procedure

5. (a) *Measurement of Indentation*.—The presser foot shall be lowered first until contact is made with the test

specimen. The indenter shall then be lowered. The points of indentation shall be at a minimum distance of  $\frac{1}{2}$  in. from the edge of the test specimen. The full load shall then be allowed to act immediately, without shock, on the indenter. Readings on the indentation dial shall be made exactly 30 sec. after the full load is applied.

(b) *Number of Readings*.—The test specimen shall be shifted to a new position after each contact in order to avoid errors due to fatigue effects. For standard tests a minimum of five readings shall be made and their average taken as the hardness measurement, except that no reading may differ by more than plus or minus 0.002 in. from the average value. Any readings known to be definitely in error shall be discarded.

(c) *Temperature*.—All tests to be truly comparative shall be conducted at the same standard temperature which shall be recorded in every case. Test specimens shall be subjected to the standard test temperature for a sufficient period of time prior to testing to insure temperature equilibrium as may be shown by constant indentation values.

(d) *Routine Tests*.—For routine testing a sufficient number of readings shall be taken to provide values checking within plus or minus 0.002 in. The results of routine tests for control purposes shall be compared only with those obtained under essentially the same test conditions with specimens of essentially the same size and shape. These results will be comparative only.

#### Report

6. *A.S.T.M. Hardness Number*.—The average indentation value determined in accordance with Section 5 shall be expressed as a whole number signifying thousandths of an inch indentation, and reported as the A.S.T.M. hardness number of the sample.

# *Standard Method of Test for*

## INDENTATION OF RUBBER BY MEANS OF THE PUSEY AND JONES PLASTOMETER<sup>1</sup>



A.S.T.M. Designation: D 531 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 531; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers the procedure for determining the indentation of rubber by means of the Pusey and Jones apparatus. This apparatus is called a plastometer and is used to measure the depth of indentation of an indenter actuated by a dead-weight load into the surface of a rubber specimen. Its action causes plastic flow adjacent to the indenter. The indentation value obtained should not be compared or confused with hardness as measured by the Standard Method of Test for Hardness of Rubber (A.S.T.M. Designation: D 314) of the American Society for Testing Materials,<sup>3</sup> since the A.S.T.M. instrument precompresses the rubber immediately adjacent to the indenter.

### Indentation Value

2. The Pusey and Jones indentation value is the distance of indentation, expressed in hundredths of a millimeter, of

a  $\frac{1}{8}$ -in. ball actuated under a load of 1 kg.

### Apparatus

3. The Pusey and Jones plastometer is shown in Fig. 1, and consists essentially of an indenter acting under a dead-weight load and a suitable device for indicating the depth of indentation. These parts shall be so mounted in a supporting frame that the indenter and weight may be independently raised or lowered vertically, permitting the indenter to rest on the surface of the test specimen and the weight to be subsequently applied to the indenter.

(a) *Support*.—The supporting frame shall consist of parallel base and top plates held apart by bolts which are threaded to carry two intermediate moveable plates. The weight is suspended from the upper moveable plate by means of a hollow tube having a shoulder which rests on the plate. The lower moveable plate shall carry on a pedestal passing through openings in the upper plates the indicator gage and also the indenter which is attached to

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

the spindle of the gage by means of the indenter shaft. This shaft shall pass freely through suitable openings in all of the plates and the weight tube. The moveable plates shall be equipped with

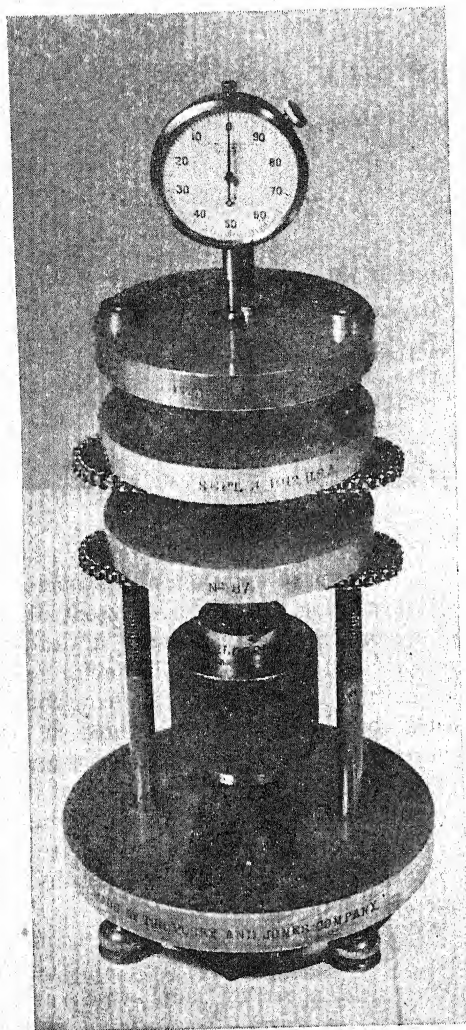


FIG. 1.—Pusey and Jones Indentation Instrument.

screw, sprocket, and chain arrangements for raising and lowering them. The base plate as supplied shall have four feet attached by ball joints permitting use of the instrument either on a flat hori-

zontal surface or on curved surfaces such as those of rubber-covered rolls. The base plate shall also be provided with an opening by which the entire instrument may be mounted on a rod and secondary base so as to allow clearance for specimens having dimensions larger than can be accommodated when using the regular feet.

(b) *Indenter*.—The indenter shall consist of a vertical steel shaft attached at the upper end to the spindle of the indicator gage and having at the lower end a steel ball point. The preferred steel ball shall be  $0.1250 \pm 0.0005$  in. in diameter and shall be made of highly polished, noncorrosive hard metal properly treated to resist wear. With very soft specimens, it may be necessary to use a ball 0.250 in. in diameter, in which case this fact shall be stated in the report. Otherwise, the indentation shall be assumed to be that of the smaller ball. The indenter shaft shall pass through the center of the loading weight and shall be provided with a suitable shoulder so that the dead-weight load may be fully applied to the ball point by proper screw adjustment of the positions of the moveable plates.

(c) *Weight*.—The weight shall consist of a metal cylinder and tube weighing  $1000 \pm 0.01$  g., placed concentric with the indenter shaft but not touching it except when resting on the shoulder during test.

(d) *Indicator Gage*.—Vertical movement of the indenter shall actuate a suitable dial gage graduated to show indentation in 0.01 mm.

(e) *Specimen Holder*.—A specimen holder for the standard test specimens described in Section 4 (a) shall be provided in which the specimens may be held flat and free from slight movements which introduce variations into the test. It consists of a clamp made of two brass

plates held together by two threaded bolts as shown in Fig. 2. The plates shall be  $4\frac{1}{4}$  in. in length,  $1\frac{1}{4}$  in. in width, and  $\frac{1}{4}$  in. in thickness. Flat, smooth holes shall be drilled  $\frac{3}{8}$  in. from each end and midway of the width. The top plate shall be provided with a hole and slot through which the indenter may act.

### Test Specimens

4. (a) The standard test specimen shall be a uniform molded rectangular

### Procedure

5. (a) *Measurement of Indentation.*—The specimen shall be placed on a firm base with the indentation surface horizontal. Flat specimens may rest on a table having a true flat surface or on the secondary base mentioned in Section 3 (a). Curved specimens such as rubber-covered rolls shall be so placed that the center-line of the indenter shaft is perpendicular to the tangent at the point of contact of the indenter. When using the standard specimen described in Section 4 (a), it shall be clamped

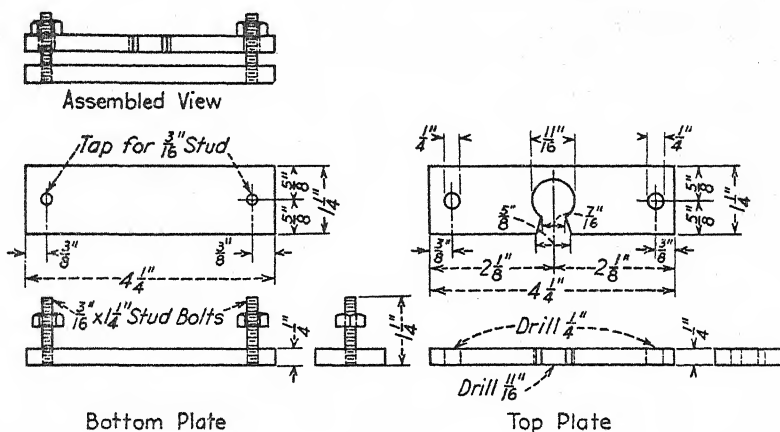


FIG. 2.—Holder for Test Specimen.

block  $\frac{1}{2}$  in. in thickness,  $1\frac{1}{4}$  in. in width, and 3 in. in length. The flat faces shall be as parallel as possible. This specimen shall be used in all cases when the apparatus is employed for reference standard purposes as distinguished from routine control testing of commercial articles.

(b) In routine testing, specimens may be used which have dimensions other than those of the standard specimen including manufactured products of such size and shape that they will support the apparatus or that it can be suitably mounted above them.

in the specimen holder with the bolts tightened sufficiently to hold it firmly but not to compress it. The apparatus shall be mounted over the specimen and adjusted, by means of a spirit level placed on the base, so that the indenter shaft is vertical. The plastometer must not rock or be unsteady whether its feet rest on the table around a small specimen or directly on a large specimen or whether it is mounted on a rod and secondary base as is required with the standard specimen and holder. The indenter shaft shall be lowered until contact is made with the specimen

and the gage needle makes three revolutions. The dial shall then be adjusted to read zero for the needle position. The weight shall be lowered onto the indenter carefully without shock and the supporting plate lowered still further so that the weight rests fully on the indenter as shown by a space of approximately  $\frac{3}{16}$  in. between the supporting plate and the shoulder of the weight tube. The amount of indentation shall be read on the gage exactly 1 min. after the application of the load.

(b) *Number of Readings.*—Three readings shall be taken, shifting the specimen to a new position for each reading.

(c) *Temperature.*—All tests to be truly comparative shall be conducted at the same temperature which shall be recorded in every case. Test specimens shall be subjected to this test temperature for a sufficient period of time prior to testing to ensure temperature equilibrium as may be shown by constant indentation values.

(d) *Routine Tests.*—When tests are made for control purposes on commercial articles of dimensions differing from

those of the standard specimen and without using the specimen holder, the results shall be compared only with those obtained under essentially the same conditions of specimen size and shape, temperature, and method of assembly of the apparatus and specimen.

(e) *Pusey and Jones Indentation Number.*—The average indentation value expressed as a whole number indicating the hundredths of millimeters of indentation shall be known as the Pusey and Jones indentation number.

### Report

6. The report shall include the following:

- (1) The Pusey and Jones indentation number,
- (2) Description of test specimen including dimensions,
- (3) Date of vulcanization, if known,
- (4) Duration and temperature of vulcanization, if known,
- (5) Size of ball point used,
- (6) Temperature of test room, and
- (7) Date of test.



# Standard Methods of Test for

## COMPRESSION-DEFLECTION CHARACTERISTICS OF VULCANIZED RUBBER<sup>1</sup>



A.S.T.M. Designation: D 575 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 575; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover two test procedures for determining the compression-deflection characteristics of vulcanized rubber compounds other than those usually classified as hard rubber and sponge rubber. The tests constitute one kind of compression stiffness measurement. The two different procedures are as follows:

*Method A. Compression Test at Specified Deflection.*—A compression test in which the load required to cause a specified deflection is determined.

*Method B. Compression Test at Specified Load.*—A compression test in which the specified weight or compressive force is placed on the specimen and the resulting deflection is measured and recorded.

### Apparatus

2. (a) *Compression Testing Machine.*—A standard compression testing machine conforming to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Desig-

nation: E 4) of the American Society for Testing Materials<sup>3</sup> and having a speed of approximately 0.5 in. per min. may be used for either type of test. Any other type machine that will meet these requirements may be used. For example, a platform scale equipped with a yoke over the platform and a hand operated screw to apply the load will serve if it will conform to the requirements prescribed for accuracy and speed. Compression tests at specified loads may be performed on a weight-deflection machine of any type that will apply specified minor and major loads gently, without impact. The machine shall be equipped to permit measurement of the deflection caused by the increase from minor load to major load.

(b) *Dial Gage.*—The deflection shall be read on a gage of dial type graduated in thousandths of an inch.

(c) *Micrometer.*—The thickness of the specimen shall be measured with a micrometer graduated to 0.001 in. having a presser foot  $0.25 \pm 0.01$  in. in diameter exerting a total force of  $3.0 \pm 0.1$  oz. The load shall be applied by means of a weight.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1940 to 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



### Test Specimens

3. (a) The test may be performed either on whole completed articles or on standard test specimens, as specified (Note).

NOTE.—Comparable results are obtained only when tests are made on specimens of exactly the same size and shape, tested to the same percentage deflection.

(b) Standard test specimens shall be  $1.129 \pm 0.005$  in. in diameter (1.000 sq. in. in area) and  $0.500 \pm 0.010$  in. in thickness, from which all molded surface layers have been removed.

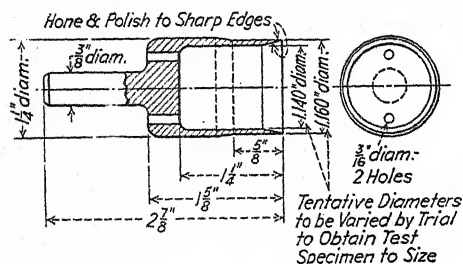


FIG. 1.—Cutting Tool.

### Preparation of Specimens

4. (a) The standard test specimens may be prepared as follows: A slab slightly over 0.500 in. in thickness may be cut from the article or molded from the same material, and ground on both sides to obtain smooth parallel surfaces and the standard thickness of  $0.500 \pm 0.010$  in. The grinding shall be carried out as prescribed in Section 9 of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials,<sup>3</sup> and shall be done without overheating the rubber. The specimens may then be cut from the slabs by means of a suitable rotating hollow cutting tool similar to that illustrated in Fig. 1. In cutting the specimen, the die shall be suitably rotated in a drill press or similar

device and lubricated with soapy water so that a smooth-cut surface having square edges is obtained. The cutting pressure shall be kept sufficiently low to avoid "cupping" of the cut surface.

(b) When whole completed articles are subjected to these tests, the surfaces which will contact the platens of the testing machine shall be cleaned of any dust, bloom, grease, or other foreign material. Grinding is not required, as this is a nondestructive test.

### Test Conditions

5. The temperature of the testing room shall be maintained between 70 and 90 F. (21 and 32 C.). The specimens to be tested shall be kept in this room for at least 30 min. prior to the time of testing.

#### *Method A. Compression Test at Specified Deflection*

### Nature of Test

6. In this method the compressive loads are applied and removed in three successive cycles. The first two cycles are for the purpose of conditioning the specimen, and the readings are taken during the third application of load.

### Procedure

7. (a) After the thickness of the test specimen has been measured, the specimen shall be placed between the platens of the testing machine. Sheets of sandpaper<sup>4</sup> shall be placed between the rubber surfaces and the testing machine platens. The sandpaper resists lateral slippage of the rubber at the contact surfaces, and should be slightly larger than the specimen. The sandpaper shall be omitted when the surface of the specimen is bonded to metal.

(b) The load shall be applied at the

<sup>4</sup> Waterproof sandpaper, 400 Grit A "Speed-Wet" manufactured by the Behr-Manning Co., or the equivalent, has been found satisfactory.

rate of approximately 0.5 in. per min. until the specified deflection is reached, after which the load shall be immediately released at the same rate. This loading cycle shall be repeated a second time. Then a small initial load (not over 1 per cent of the amount required in the first two cycles) shall be applied and the deflection gage set at zero, after which the load shall be applied as before until the specified deflection is again reached. The load required shall then be read and recorded.

(c) If desired, the machine may be stopped at intervals of 5 per cent deflection during the third application and the load recorded for each deflection so that a stress-strain curve may be drawn.

(d) Percentages of deflection shall be based on the thickness of the specimen prior to the first load application.

#### *Method B. Compression Test at Specified Load*

##### **Nature of Test**

8. This test is intended for rapid testing with a weight-deflection type of machine, although it can be performed on the other machines described in

Section 2 (a). Because speed with reasonable accuracy is desired, a single load application cycle is used.

##### **Procedure**

9. A specified minor weight or force shall be applied for a period long enough to adjust the deflection gage, after which the major load shall be applied for 3 sec. The deflection shall be read on the dial gage (Section 2(b)) at the end of the 3-sec. period, and shall not include any deflection caused by the minor load. The percentage of deflection shall be calculated on the basis of the original thickness of the specimen.

##### **Report**

10. The report shall include the following:

- (1) The deflection expressed as a percentage of the original thickness of the specimen,
- (2) The load in pounds per square inch of original cross-section area,
- (3) Description of sample and type of test specimen, including dimensions,
- (4) Description of test apparatus,
- (5) Temperature of test room, and
- (6) Date of test.

*Standard Methods of*  
DYNAMIC TESTING FOR PLY SEPARATION AND CRACKING  
OF RUBBER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 430 - 40

ADOPTED, 1940.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 430; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These methods are intended for use in estimating the comparative ability of soft rubber compounds to resist dynamic fatigue. They do not apply to the testing of the material ordinarily classed as hard rubber. They are not suited for use in purchase specification requirements both because correlation with service life is uncertain and because the results from duplicate specimens do not check with sufficient precision. No relation between the results of the tests and actual service performance is given or implied. While not accurate measures, they do, however, yield data on which to base a judgment as to service quality and are widely used in research and development work. When properly applied and interpreted, the tests give comparative results of unquestioned value.

#### Types of Tests

2. (a) Tests for dynamic fatigue are designed to simulate the continually re-

peated distortions received in service by many rubber articles, such as tires, belts, footwear, and various molded goods. These distortions may be produced by extending, compressing, and bending forces or combinations of them. The effect of the distortions is to weaken the rubber until surface cracking or actual rupture occurs. In the case of combinations of rubber with other flexible material such as fabric, the effect may be evidenced by separation between the materials, caused either by breaking of the rubber or failure of the adhesion or both.

(b) These tests are, therefore, of the following two types:

*Type I.*—Tests designed to produce separation of rubber-fabric combinations such as are used in belts and tires, involving controlled bending of the specimens.

*Type II.*—Tests designed to produce cracking on the surface of rubber by either repeated bending or extension as occurs in service with parts such as tire treads and sidewalls, rubber soles, and shoe uppers, diaphragms, hose covers, etc.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1935 to 1940, being revised in 1940.

(c) These methods cover three test procedures using the following different types of apparatus:

Method A. Scott Flexing Machine.

Method B. De Mattia Flexing Machine.

Method C. E. I. du Pont de Nemours and Co. Flexing Machine.

The Scott flexing machine is used principally for tests of type I, the De Mattia flexing machine for tests of type II, while the du Pont apparatus is adapted to tests of either type I or II.

#### Application

3. In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular material, the latter shall take precedence.

#### Preparation of Sample

4. Except as may be otherwise specified in these methods, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials<sup>3</sup> shall be complied with and are made a part of these methods.

#### METHOD A. SCOTT FLEXING MACHINE<sup>4</sup>

##### Type of Flexure

5. The Scott flexing machine method is used to test for ply separation samples of belts, tires, or other articles composed of plies of fabric bonded together by rubber compounds.

#### Test Specimens of Belts

6. Test specimens selected from samples of belts shall be cut lengthwise of the belt and their locations recorded. The seam area of a folded belt shall not be included in any of the test specimens and the folded edge shall be removed before cutting the specimens. The specimens shall consist of four plies for routine tests, any excess plies being removed by stripping carefully so as not to weaken the remaining bonds. The specimens shall be 8.250 in. in length by 1 in.  $\pm$   $\frac{1}{16}$  in. in width. The actual thickness shall be measured and recorded.

#### Test Specimens of Tires

7. Suitable test specimens cannot be cut from finished tires and it is necessary to prepare special flexing pad samples as described in the following Paragraphs (a) to (c) from the cord fabric and rubber compounds which are to be tested:

(a) *Building Unvulcanized Flexing Pad Sample.*—Solutioned, frictioned, or bare cord fabric shall be calendered with the rubber compound to a total thickness of 0.050 in. Six plies of this material shall be assembled using a hand roller so that the plies run in alternate directions. The first, third, and fifth plies shall have the cords lengthwise and the second, fourth, and sixth plies crosswise of the pad. Care shall be taken that the same calendered side of each piece is facing up and that each alternate ply crosses at right angles. The pad which then has a thickness of 0.300 in. shall be cut by means of a template and knife to dimensions of 4.94 by 7.94 in. The long edge of the template shall be held parallel with the lengthwise cords in the pad.

(b) *Vulcanizing of Flexing Pad Samples.*—The flexing pad sample shall be vulcanized in a steel mold having a cavity measuring 5 by 8 by 0.325 in. Uniform compression shall be applied

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> Method A was originated by General Laboratories, U. S. Rubber Co. For further information respecting this test see W. A. Gibbons, "Flexing Test for Tire Carcass Stocks," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 2, No. 1, p. 99, January 15, 1930; also W. L. Sturtevant, "Rubber Power Transmission Belting, Part III—Flexing Machine and Dynamometers for Testing Belting Quality," *India Rubber World*, Vol. 83, No. 3, p. 67 (1930).

over the entire top surface of the pad. This compression, together with slight stretching produced by the unvulcanized pad being cut slightly smaller than the cavity, ensures straight cords in the cured pad. In order to obtain the compression it is necessary to make up the difference between the pad thickness and the mold depth by means of filler layers of holland cloth or tin foil which are placed on top of the pad. These filler layers shall be added until the total thickness of the assembly, measured by means of a dial micrometer gage using a little pressure with the fingers on both sides of the gage foot, is 0.305 in. A sheet of rubber compound containing curing ingredients and measuring 6 by 9 by 0.02 in. shall be placed on top of the flexing pad and filler layers over the cavity of the mold before the mold cover is placed in position. The total thickness of the material in the mold is then 0.325 in. and expansion will produce the correct pressure to make a compact, undistorted pad. The purpose of the top rubber layer is to fill the overflow space and seal the mold. In placing the flexing pad in the mold, care shall be taken to keep uppermost that side of the pad having the cords running crosswise. Vulcanization shall be carried out by heating the mold under pressure at a known temperature for the required time in a rubber vulcanizing press. After curing, the filler layers shall be removed and the pad allowed to cool and rest at a room temperature between 70 and 90 F. (21 and 32 C.) for not less than 36 hr. before being tested. Pads made in this manner shall be 8 in. in length by 5 in. in width and shall have a thickness of 0.275 to 0.280 in. Any pads having wrinkled cords shall not be tested.

(c) *Cutting Tire Test Specimens.*—Four strips each 8 in. in length by 1 in. in width shall be cut from the tire

flexing pad. On the first cut, a strip 0.250 to 0.500 in. in width from the edge along the long side of the pad shall be removed and discarded. Care shall be taken to cut the strips straight, with smooth edges and as closely to the exact width as possible. If reasonable care has been taken in preparing the pads and in cutting the specimens, there

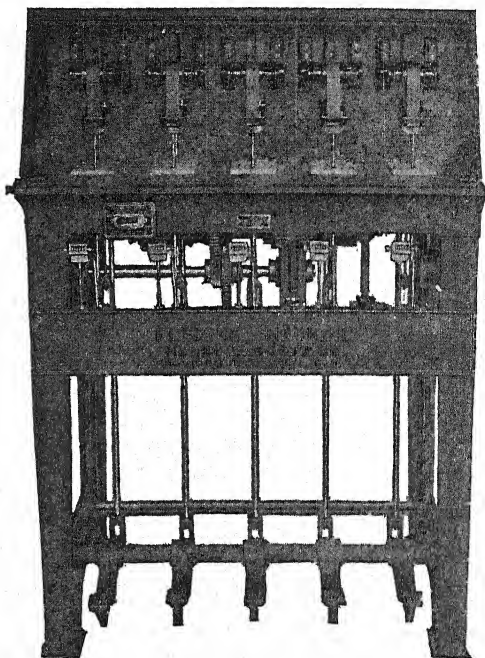


FIG. 1.—Scott Flexing Machine with Five Hubs.

should not be more than five or six cut longitudinal threads showing on the two edges of a six-ply specimen. An excessive number of such cut threads indicates that many of the threads of alternate plies are not parallel.

#### Number of Test Specimens

8. At least five specimens from each belt sample or four specimens from each tire pad shall be tested and the results averaged. The accuracy of the final value may be increased by testing a greater number of specimens.

### Scott Flexing Machine

9. The essential features of the apparatus, illustrated in Fig. 1, are as follows: The flexing machine has five hubs and is capable of testing five specimens at one time. Each hub is carried on a loading lever and rotates on suitable bearings. The test specimens shall be bent around the hubs having an arc of contact of about 135 deg. and the ends shall be gripped by clamps which are oscillated up and down by rocker arms driven through a chain of gears by a  $\frac{1}{4}$ -hp., 1750 r.p.m. motor. The action on the specimen is a flexing back and forth over the hub while pulled taut by the loading lever and weight. The specimen has a travel in one direction of 2.625 in. and a full cycle travel of 5.250 in. The speed of operation is approximately 160 cycles per minute, the exact number of cycles in each test being recorded on a counter affixed to each rocker arm. The entire machine is approximately 50 in. in height and occupies a floor space of about 26 by 38 in.

### Hub Size and Flexing Load

10. Specimens of belts shall be tested using hubs 1.250 in. in diameter and a 100-lb. flexing load. Specimens of tire cord shall be tested using hubs 0.563 in. in diameter and a 100-lb. flexing load.

### Procedure for Belts

11. The test specimens of belts shall be bent around the hubs with the pulley side of the belt against the metal and the ends clamped in the grips. The flexing load shall be then carefully applied without shock, the counter set at zero, and the machine started. It shall be allowed to run until some fine particles dislodged by friction may be seen on the white plate beneath the hub which indicates that separation of the plies has started. Frequent inspection

of the specimens undergoing test is imperative if reliable results are to be expected. When the first indication of ply separation appears, the counter reading shall be noted and thereafter the specimen shall be more closely watched and the frequency of the inspection increased to insure proper determination of the end point. When there is a clear separation across the width of the specimen it shall be considered to have failed. The minimum counter reading for this failure shall be recorded as the end point. Notation shall also be made as to the location of the separation. When a test is started, it shall be continued to completion without interruption. The machine shall not be stopped and allowed to remain inactive for any length of time and then started again. However, for examining the specimen, each hub may be released momentarily from its weight by means of the foot lever provided. None but experienced operators should make this determination.

### Procedure for Tires

12. The test specimens for tires shall be mounted with the lengthwise outer ply cords against the hub of the machine and shall be tested in a manner similar to the procedure for belts (Section 11). In the case of tire specimens, after the specimen has been run about 10 min. a thick coat of molten caranauba wax shall be brushed on the outer side of the specimen at the flexing region. As soon as separation begins, the temperature of this section increases very rapidly and the wax melts. The melting of the wax starts with a small area and gradually spreads as separation increases. This serves as a warning that complete separation will occur shortly thereafter and eliminates the necessity of examining the specimen at frequent intervals during the early part of the test. The interval

between the melting of the wax (the time at which separation actually starts) and complete separation across the specimen may not always be the same (Note).

NOTE.—A specimen with a short flexing life will show complete separation soon after the wax melts, whereas, a sample with a greater flexing life might require a time interval two or three times as long. However, with specimens having similar flexing life, the time interval between the melting of the wax and complete separation is fairly constant.

### Calculations

13. The result of the test of any sample shall be calculated as the average of the number of flexing cycles required to produce complete separation of each test specimen as determined from the counter readings.

### METHOD B. DE MATTIA FLEXING MACHINE<sup>5</sup>

#### Type of Strain

14. The De Mattia flexing machine method may be used to test rubber specimens for resistance to cracking produced either by extension or bending, depending on the relative adjustment of the stationary and movable grips, and the distance of travel of the latter. The choice of type of strain is optional (Note) but notation shall be made of the type actually used, giving full details of the relative positions of the grips and of the travel.

NOTE.—In choosing the type of strain, it should be remembered that the phenomenon of cracking starts on the surface of the rubber and rapidly progresses inward as new surface is exposed. Since rubber is practically noncompressible but highly extensible, the rupture of the surface fibers in both types of strain must come from disturbances due to elongation. The magnitude of the extension,

however, may differ and the internal distribution of force in the specimens is not the same in the two cases. The choice, therefore, will depend considerably on the purpose of the test and the kind of service for which correlation of the test results may be sought.

### Test Specimens for Extension Fatigue Cracking

15. When the extension type of strain is used the standard test specimen shall be the dumbbell-shaped tension specimen shown in Fig. 3 (die C) of the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials.<sup>3</sup> The actual thickness shall be measured and recorded and results shall be compared only when obtained using specimens of substantially

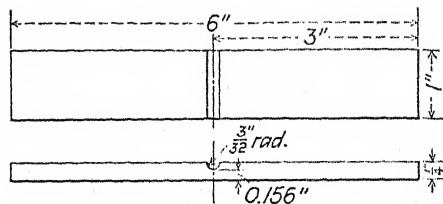


FIG. 2.—De Mattia Bend Flexing Specimen with Circular Groove.

the same thickness. The specimens shall be prepared in the same manner as for tension tests and special care shall be taken to avoid any surface imperfections which might start the cracking. All specimens with irregularities on any surface shall be discarded. On specimens cut from molded sheets, highly polished surfaces are very desirable. When buffing is necessary, as with samples cut from finished products, great care shall be exercised that the buffing is alike on all specimens which are to be compared.

### Test Specimens for Bend Flexing

16. When the flexure is by bending, special molded specimens of the shape

<sup>5</sup> See L. V. Cooper, "Laboratory Evaluation of Flex-Cracking Resistance," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 2, No. 1, p. 391, October 15, 1930.



shown in Fig. 2 shall be used. The specimens shall have highly polished surfaces and shall be free from surface irregularities which might start the cracking prematurely. The minimum thickness in the circular groove shall be measured in thousandths of an inch and recorded. Results shall be compared only between specimens having thicknesses agreeing within plus or minus 0.0015 in.

#### Number of Test Specimens

17. At least three specimens from each sample shall be tested and the

lar reciprocating member for holding the other end of each of the specimens. The reciprocating member is so mounted that its motion is straight in the direction of and in the same plane as the center line between the grips. The travel of the moving member shall be adjustable and shall be obtained by means of a connecting rod and eccentric having a minimum length ratio of 10 to 1. The eccentric shall be driven by a motor operating at constant speed under load and giving 300 flexing cycles per minute. Provision shall be made for a maximum travel of the moving grips of

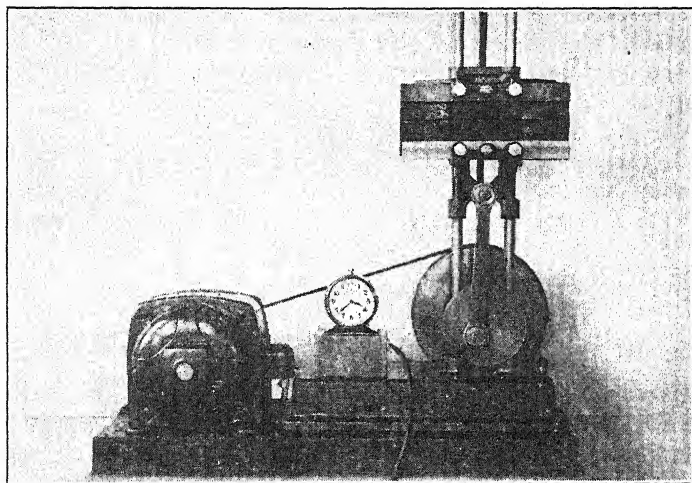


FIG. 3.—De Mattia Tester with Time-Switch for Starting or Stopping, Arranged with Specimens for Flex-Cracking Test.

results averaged. It is desirable, when possible, to test simultaneously with each set of specimens a set of control specimens of which the resistance to flex cracking is known.

#### De Mattia Flexing Machine

18. The essential features of the apparatus, one design of which is shown in Fig. 3, are as follows: The machine has an adjustable stationary head or member provided with suitable grips for holding one end of each of the test specimens in a fixed position and a simi-

lar reciprocating member for holding the other end of each of the specimens. The capacity of the machine shall be such that tests at the same time may be made on at least six and preferably twelve specimens. The grips shall hold the specimens firmly throughout the test and those on the reciprocating member may clamp each specimen individually to facilitate proper adjustment of the specimens.

#### Clamping Specimens in Machine

19. One end of the specimen shall be clamped in the stationary grip and the other in the movable grip, care being



taken to see that the long axis of the specimen is parallel to the direction of motion. The constricted section or the circular groove of the clamped specimens shall be located symmetrically midway between the clamps. The specimens for extension fatigue cracking shall be gripped only on the enlarged ends. The specimens may be mounted on the machine most conveniently by holding them properly spaced in parallel positions in a special rack. The distance between the outer edges of the side bars of the rack shall be equal to the space between the jaws of the testing machine when positioned for holding the specimens without tension. The specimens can be mounted on the testing machine by bringing the jaws into contact with the mounting rack and tightening the clamps on the projecting ends of the specimens. In the case of the specimens for bend flexing, the clamps shall grip each end over a length of  $1\frac{3}{8}$  in. with the side having the circular groove so placed that it will be the outer surface when the specimens are bent.

#### Adjustment of Machine

20. (a) *Extension Fatigue Cracking*.—For these tests, the positions of the stationary and movable grips relative to each other and the length of the eccentric arm and connecting rod shall be adjusted by trial so that the movable grip will approach the stationary grip  $\frac{1}{2}$  in. closer than necessary to relieve the elongation stress in the specimen and so that the grips will separate a maximum distance sufficient to elongate the portion of the specimen between the gage marks a predetermined and recorded amount. The elongation of the specimens between the gage marks shall not exceed one fourth of the ultimate breaking elongation. For highly extensible rubbers a maximum elongation of 125 per cent is suitable.

(b) *Bend Flexing*.—For bend flexing tests the positions of the stationary and movable grips relative to each other and the length of the eccentric arm and connecting rod shall be adjusted so that during each stroke of the machine the grips approach each other to a distance of  $\frac{3}{4}$  in. and separate to a distance of 3 in.

#### Procedure

21. After adjustment of the apparatus and specimens is completed, the machine shall be started and the time recorded. The test shall be continued until, by frequent inspection, the appearance of the first minute sign of cracking is detected, when the time shall again be recorded. The first cracking may be evidenced as either very fine hair-line cracks or as slight pin holes. After this time, the specimens shall be observed very closely until the test is discontinued and the final time recorded when the cracks have developed sufficiently to permit grading the degree of the cracking in all specimens as described in Section 22. It is not desirable to run the specimens until actual complete rupture occurs when this can be avoided. When testing specimens of which the dynamic fatigue properties are approximately known, the test may be run for a known predetermined number of cycles after which the grading comparison shall be made.

#### Results

22. After conclusion of the test, the specimens shall be removed from the machine and evaluated in sequence relative to the seriousness of the cracking by comparison with a standard scale of cracked specimens of the same type. The comparison shall be made by judging visually the length, depth, and number of cracks. The standard comparison scale shall consist of ten specimens equally graded and numbered from

No. 0, showing no cracking, to No. 10, which is completely cracked through. The final result for a given sample shall be recorded as the average of the numbers so obtained from all of the specimens. The number of cycles required for the first sign of cracking and for the final result shall be calculated by multiplying the observed time expressed in minutes by the rate of 300 cycles per minute and shall also be recorded. The test results shall be compared only

method A and described in Sections 6 and 7 (a), (b) and (c), except that they shall be cut to dimensions of 1 in. in width by 4 in. in length.

#### Test Specimens for Flex Cracking

24. Specimens for tests of type II (Section 2 (b)) shall be specially prepared from the unvulcanized rubber compounds to be tested. They shall have a fabric base to prevent stretching during test and shall be strips 1 in. in

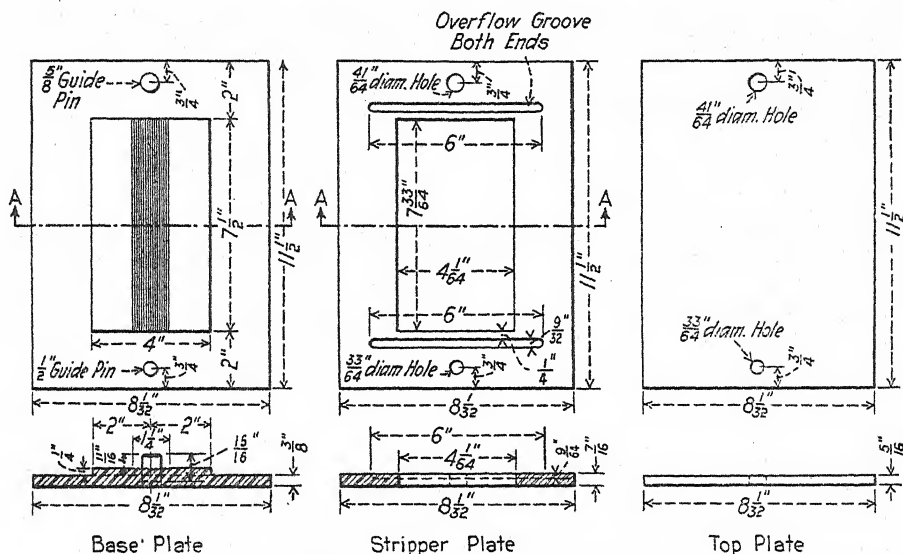


FIG. 4.—Mold for Preparing Test Specimens for du Pont Flexing Test.

between specimens of similar sizes and shapes, tested in the same manner under identical conditions.

#### METHOD C. E. I. DU PONT DE NEMOURS AND CO. FLEXING MACHINE<sup>6</sup>

##### Test Specimens for Ply Separation

23. Specimens for tests of type I (Section 2 (b)) shall be prepared as in

<sup>6</sup> Method C was originated by Fisk Rubber Co. Laboratories. For further information respecting this test see A. M. Neal and A. J. Northam, "Some Factors Affecting the Resistance to Flexing," *Industrial and Engineering Chemistry*, Vol. 23, No. 12, p. 1449, December, 1931.

width by 4 in. in length cut at right angles to the grooves from vulcanized test slabs prepared as described in the following Paragraphs (a) and (b):

(a) The unvulcanized stock shall be prepared in sheets having a thickness of  $0.185 \pm 0.003$  in. A slab 4 by 7 1/2 in. shall be cut so that the grain of the stock runs parallel to the 4-in. side. The slab shall then be backed with a layer of frictioned belt fabric cut to the same size. The warp of the fabric shall be placed parallel to the grain of

the stock. Semicured white letters may be placed along the side of the slab for the identification of the samples, which shall be subsequently cut out and assembled for flexing. Before vulcanizing, the stock shall be allowed to rest 16 hr. after mixing. The slab and fabric shall conform to the following requirements:

Stock thickness.....	0.185 ± 0.003 in.
Weight of slab (1.15 sp. gr.), approximate.....	105 g.
Weight of fabric.....	25 g.
Total weight.....	130 g.
Overflow, approximate.....	15 g.

	Warp		Filler
<i>Raw Fabric, Silver Hard Duck:</i>			
Threads per inch....	27½	by	18½
Ply.....	7	by	4
Crimp.....	25	by	4 per cent
Strength (A.S.T.M. Grab Method), <sup>a</sup>			
12-hr. exposure in an atmosphere having a relative humidity of 65 per cent at 70 F.....	600	by	290 lb.
Thickness.....			0.057 in.
Weight per square yard.....			28 oz.
<i>Fricioned Fabric (fricioned both sides):</i>			
Weight per square yard.....			36 oz.
Thickness.....	0.049		± 0.003 in.

<sup>a</sup> The grab method is described in Section 10 of the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.<sup>2</sup>

(b) The stretch is not taken out of the fabric, but this is of little importance since there is little stretch to silver hard duck. The fabric shall be fricioned on both sides with a conventional gum-friction compound. The slab shall be placed in the mold shown in Fig. 4 with the rubber side of the slab next to the mold corrugations and vulcanized as required for the particular compound. This produces a cured slab with a fabric backing 7.5 in. in length by 4 in. in

width and 0.188 in. in thickness at the smooth portion and 0.233 in. in thickness at the corrugated portion. There are eight transverse V-shaped grooves 0.156 in. ( $\frac{5}{32}$  in.) in width and 0.045 in. in depth. The angle of the "V" is 120 deg. Notation shall be made of the time and temperature of vulcanization.

### Number of Test Specimens

25. At least five specimens from each sample shall be tested in ply separation tests or at least three specimens from each test slab in flex cracking tests and the results averaged. The accuracy of the final value may be increased by testing a greater number of specimens. Since the test is primarily comparative, a set of control specimens from samples of which the qualities are known should be tested simultaneously whenever possible. Flex cracking results shall be compared only between specimens having thicknesses agreeing within plus or minus 0.0015 in.

### du Pont Flexing Machine

26. The du Pont flexing machine, shown in Fig. 5,<sup>7</sup> consists essentially of four pulleys around which a test belt is run. The belt shall be composed of test specimens acting as links held together with belt fasteners. The machine may be arranged as shown so that several separate belts made of test specimens may be run at the same time. Facing the front of the machine, the upper left-hand pulley is driven by a  $\frac{1}{2}$ -hp. motor. This driving pulley shall have a speed of 930 r.p.m. The upper right-hand pulley is on a balance arm which supports a 30-lb. weight. The weight is 11 in. and the pulley 5 in. from

<sup>7</sup> Blueprints of detailed drawings for the construction of this apparatus are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

the supporting pin, producing a belt tension of approximately 17 lb. The center and lower pulleys are idlers. All pulleys are 3 in. in outside diameter and have faces  $1\frac{1}{2}$  in. in width with 0.031 in. ( $\frac{1}{32}$  in.) crowns and are mounted on ball bearings. The bottom and center pulleys are on the same vertical line and their centers are  $14\frac{3}{4}$  in. apart. The left-hand, or driving pulley, and the

determine when a belt breaks, the lever arm contacts a switch which stops an electric clock.

#### Procedure

27. Twenty test specimens shall be formed into a test belt by means of belt fasteners<sup>8</sup> using six clips on one end of each specimen and five on the other. The belt fasteners shall be joined to-

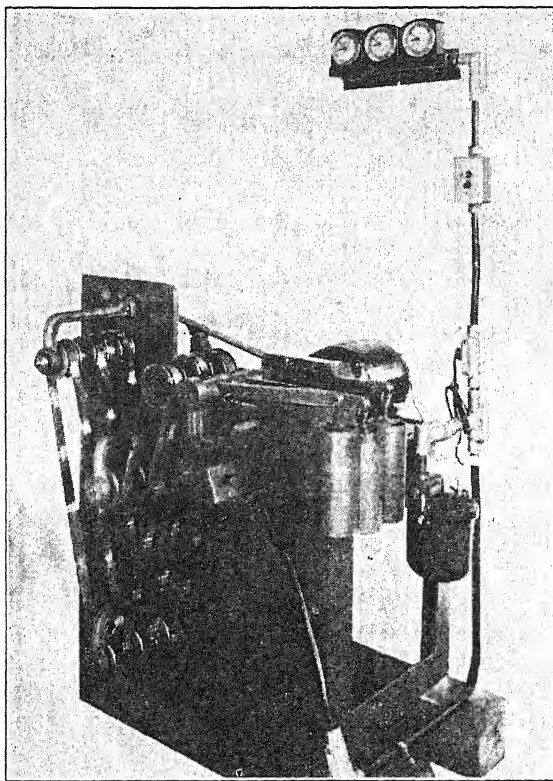


FIG. 5.—Du Pont Flexing Machine.

right-hand, or balance pulley, are located 6 in. on either side of the center pulley and 9 in. above it. The specimen belt runs over the driving pulley, under the center pulley, over the right-hand, or balance pulley, and under the bottom pulley back to the driving pulley. The angle of approach to the pulleys is 14 deg. from the vertical. In order to

gether with raw-hide pins each 1.125 in. in length. If the number of specimens to be tested is insufficient, blanks of the same size as the specimens may be used. When completed, the belt should measure about 90 in. in circumferential length. The belt shall be

<sup>8</sup> Detroit No. 5 belt fasteners are satisfactory.

placed on the pulleys and driven at a speed of 97 r.p.m. or a linear speed of of approximately 8.75 m.p.h. Belts for ply-separation tests shall have the same side of the specimens next to the drive pulley in all cases and when the specimens are cut from rubber belting, this side shall be the pulley side of the sample. Belts for flex-cracking tests shall have the fabric base next to the drive pulley. Each specimen shall be flexed three times with the face under tension and once under compression during each revolution. The direction of rotation of the belt shall be changed every 12 hr. since this gives greater uniformity. Notation shall be made of the time of starting the machine. Flexing shall be continued and visual inspection of the specimens shall be made at periodic intervals until all specimens show some signs of failure when the test shall be discontinued and the time recorded. With ply-separation specimens, notation shall be made of the time of appearance in each specimen of the first indication of ply separation visible upon bending the specimen. Failure of ply-separation specimens shall be accepted when there is clear separation across the width of the specimen. With flex-cracking specimens, the failure is indicated by the appearance in the corrugations of small nicks or pin holes which soon increase in size until they become deep cracks, which may extend all the way across the specimen. The frequency of inspection of the specimens shall be sufficient to give a reliable measure of the failure of the specimen. If a specimen breaks prematurely, it shall be replaced with a blank and the test continued.

#### Results

28. (a) *Ply-Separation Tests*.—With ply-separation specimens, the result of

the test for a given sample shall be calculated as the average of the total number of belt revolutions required to produce complete separation of each test specimen. Notation as to the location of the separation shall also be recorded.

(b) *Flex-Cracking Tests*.—With flex-cracking specimens, the result of the test shall be evaluated as given in Section 22, except that the number of flexures shall be recorded in terms of total belt revolutions at the end of the test calculated by multiplying the observed flexing time expressed in minutes by the belt speed of 97 r.p.m. A detailed permanent record of the cracking in each specimen may be made by indicating on a suitable form the location, number, and intensity of the nicks and cracks. Dots may be used for nicks and straight lines for cracks. Very light marks may be used for first indications with heavier marks to indicate increased depth and width of failure.

#### REPORT

##### Report

29. For each of the three alternative methods of test, the report shall include the following:

(1) The results of the test expressed in accordance with Section 13, 22, or 28,

(2) Statement of the purpose of the test and the method used, including a description of the specimen,

(3) All observed and recorded data,

(4) Description of the sample,

(5) Date of manufacture or vulcanization, if known,

(6) Date of test, and

(7) Temperature of the test room

# Standard Method of Test for

## YOUNG'S MODULUS IN FLEXURE OF NATURAL AND SYNTHETIC ELASTOMERS AT NORMAL AND SUBNORMAL TEMPERATURES<sup>1</sup>



A.S.T.M. Designation: D 797 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 797; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method of test covers the procedures for determining Young's modulus (in flexure) of natural and synthetic elastomers at normal and subnormal temperatures.

(b) Increases in Young's modulus of elastomers upon exposure to progressively lower temperatures are attributable to either or both of the following two distinct freezing effects (see Explanatory Note):

(1) *Second Order Transition (Vitrification)* which is relatively independent of time of exposure to low temperatures, and

(2) *Crystallization* or other effects which are strongly dependent on time of exposure to low temperature.

Procedures for measuring the effects of these two changes are different and are described separately in this method.

### Apparatus

2. The apparatus, a satisfactory ex-

ample of which is shown in Fig. 3, shall consist of the following (Notes 1 and 2):

(a) *Cooling Chamber*.—A cooling chamber of sufficient size to contain the test apparatus with openings or ducts appropriately placed for remote control and capable of attaining and holding the desired test temperatures. All specified temperatures shall be maintained within plus or minus 1.0 F., preferably by automatic regulation. Uniformity of temperature throughout the cooling chamber should be assured by means of a suitable air-mixing fan.

(b) *Supports for Specimens*.—Supports for the simple beam test specimens made in the form of a saddle as shown in Fig. 1. The curved surfaces of the supports shall be cylindrical with a radius of  $0.187 \pm 0.003$  in. and at least 1.01 in. in length. The distance between the center lines of the specimen supports shall be  $2.000 \pm 0.002$  in. Retaining pins shall be placed at each end and side of the specimen supports as shown in Fig. 1, to assist in correctly aligning the specimens and to prevent accidental dislodging of test specimens.

(c) *Liquid Containers*.—A means for completely immersing in a liquid the

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

specimen supports described in Paragraph (b). All referee or normal tests shall be made in a gas (air, carbon dioxide, or nitrogen) atmosphere, but where required by particular service conditions, tests may be run in the standard manner submerged in the specified liquid.

(d) *Loading Foot*.—The surface of the cylindrical loading foot which contacts the specimen shall have a radius of  $0.187 \pm 0.003$  in. and, like the saddle supports, a length of not less than 1.01 in. The

(f) *Loading Method*.—A means for raising and lowering the loading foot, and for applying the required load. Dead weights of appropriate size are satisfactory.

(g) *Deflection Indicator*.—A dead weight, dial micrometer gage calibrated to 0.001 in. mounted directly over the top end of the loading rod.

(h) *Dead Load Counterbalance*.—If the total dead load (loading foot, loading rod, weight pan, and dial gage reaction) exceeds 0.11 lb., a counterbalance system

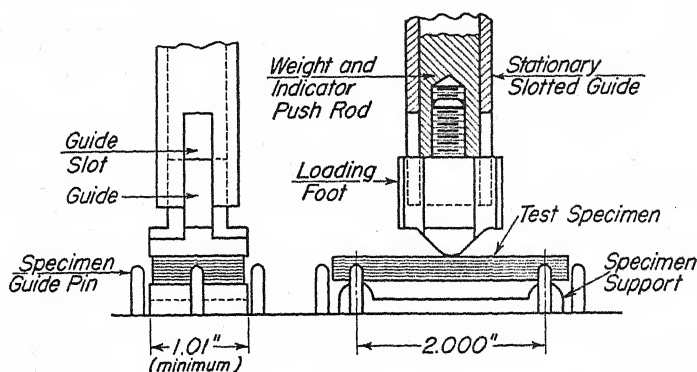


Fig. 1.—Loading Foot and Specimen Support for Low-Temperature Modulus in Flexure Apparatus.

loading foot shall be aligned with its cylindrical axis parallel to those of the saddle supports and shall contact the specimen midway between the saddle supports.

(e) *Loading Rod*.—A loading rod, shall be attached to the loading foot and shall extend outside the cooling chamber. The bearing in which the loading rod slides (whether it be inside or outside the cooling chamber) shall be as nearly friction-free as possible. (Polished rods and tubes machined from canvas-base phenolic plastic laminates and hand rubbed and lapped with graphite are recommended.)

supported on agate V's and hardened steel knife edges shall be attached to the loading rod in order to reduce the total dead load to 0.09 to 0.11 lb. See Fig. 2 for one design of a counterbalance system.

(i) *Turntable and Registering System*.—A means for arranging a number of specimen supports on a turntable or other indexing mechanism. Means for indexing each specimen in turn beneath the loading foot from outside the cooling chamber shall also be provided.

NOTE 1.—In cases of consumer specification or where apparatus such as described in Section 2 is not available, the durometer test may be used



as a secondary or emergency method of evaluating cold resistance. In such cases the durometer shall be used as specified in the Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

NOTE 2.—All metallic parts should be made of corrosion-resistant materials or should be suitably plated to avoid corrosion.

### Test Specimens

3. (a) The test specimens shall be

that a load of not more than 2.5 lb. shall be needed to produce the deflection required in Section 4 (b). The following dimensions within the tolerances indicated, will usually be satisfactory:

Width . . . . . 1.00  $\pm$  0.01 in.

Thickness . . . . . 0.250 in. uniform over the surface  
to  $\pm$  0.002 in.

Length . . . . . 2.50  $\pm$  0.025 in.

(b) Two specimens of each sample shall be tested.

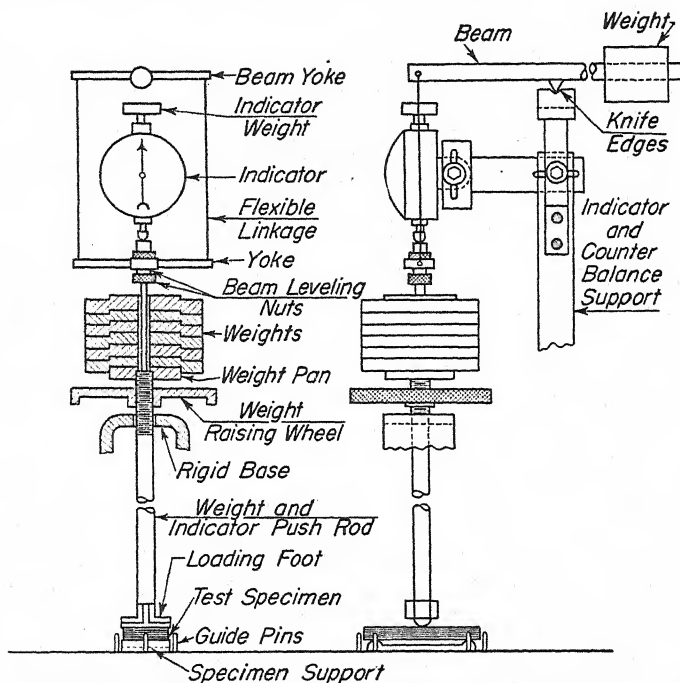


FIG. 2.—Counterbalance Device for Low-Temperature Modulus in Flexure Apparatus.

strips cut or buffed from molded sheets or they shall be molded to size from the material to be tested. They shall have a rectangular cross-section and shall be cut with their longitudinal axis parallel to the calender grain, unless grain effects are to be evaluated. The thickness of the specimen to be used will depend on the modulus of the material. Generally, the thickness shall be chosen so

**Procedure (For determination of Young's modulus - temperature relation when crystallization or other time effects, if present, are negligible.)**

4. (a) The dimensions of the test specimens shall be measured and recorded and the specimens carefully centered on the saddles. With the refrigerator controls set at 70 F., the air circulating fan shall be started (Note). The temperature

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



shall be allowed to remain at 70 F. for 15 min. before proceeding with the test.

NOTE.—It is suggested that the cooling chamber be kept tightly closed at all times during a given test, except when the inside temperature is above the existing dew point temperature.

(b) By means of the indexing and aligning mechanism the first specimen

indicator reading recorded as  $R_1$ . The load shall be reapplied, the time noted and 15 sec. later the dial indicator reading recorded as  $R_2$ . The loading foot shall be raised clear of all the specimens and the next specimen placed in position by the indexing mechanism.

(c) The test procedure prescribed in Paragraph (b) shall be repeated on all

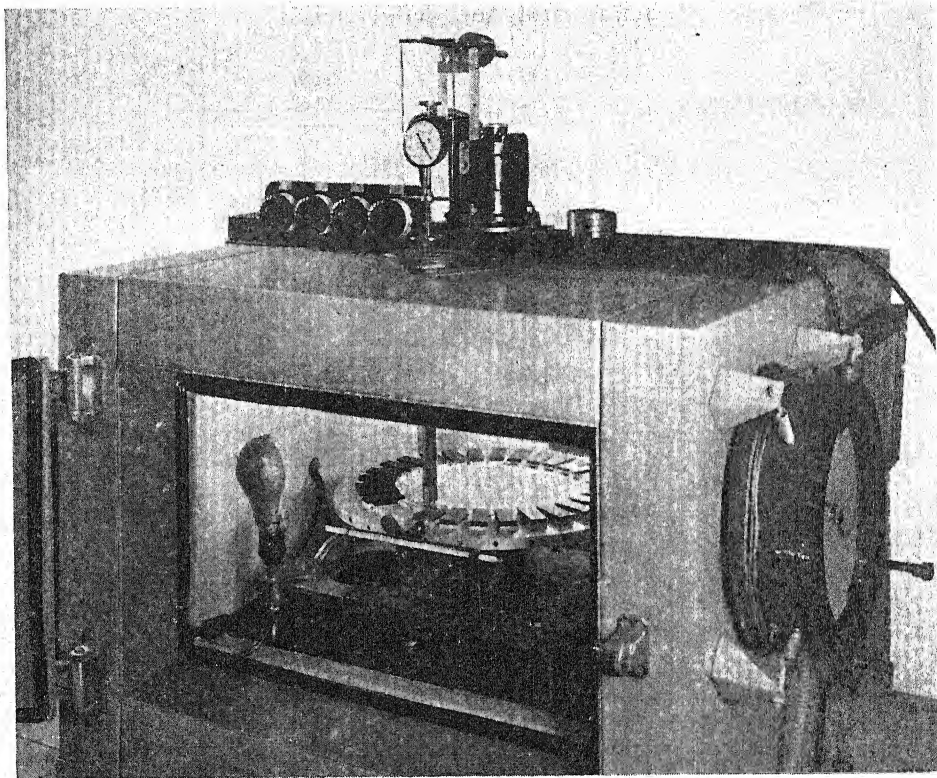


FIG. 3.—Modulus in Flexure Test Apparatus.

shall be located under the loading foot. The loading foot shall be lowered with an initial load of 0.09 to 0.11 lb. net on the center of the specimen and the dial indicator reading noted. The weight pan shall be loaded to produce a deflection of 0.010 to 0.025 in. and the specimen allowed to remain loaded for 10 sec. The weights shall then be removed and after 10 sec. the dial in-

the specimens within the chamber at 70 F.

(d) The test procedure prescribed in Paragraph (b) shall then be repeated at temperatures of +32 F., -40 F., -70 F., and a still lower temperature, if possible. Data shall also be recorded at convenient temperature intervals (10 F.) from the lowest temperature to +70 F. (increasing temperature).

NOMOGRAM FOR CALCULATING  
YOUNG'S MODULUS FROM  
SIMPLE BEAM EXPERIMENT

$$\text{EQUATION } E = \frac{Ll^3}{4wt^3(R_f R_s)}$$

WIDTH OF BEAM (w) 1 INCH  
DISTANCE BETWEEN SUPPORTS (l) 2 INCHES

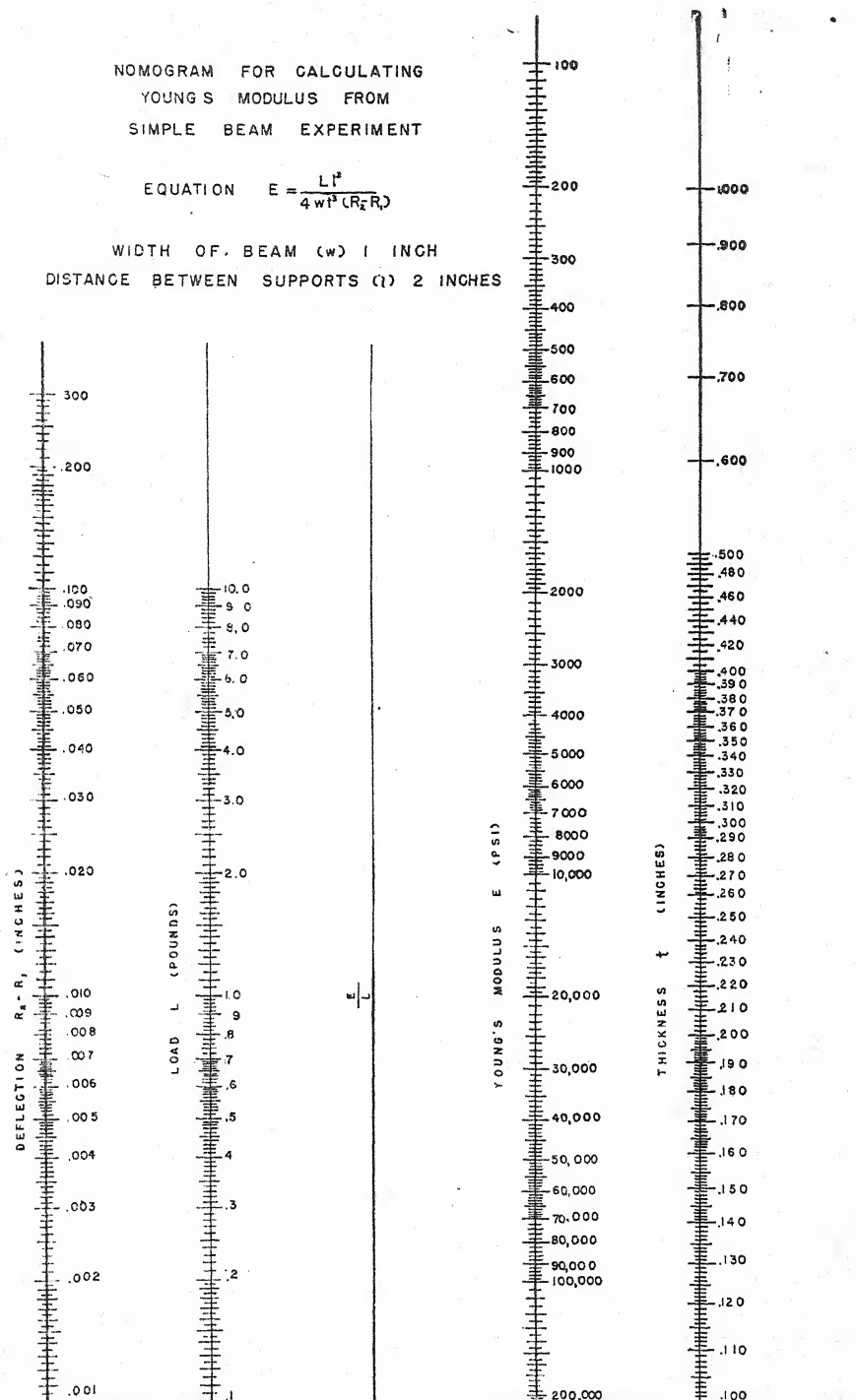


FIG. 4.—Nomograph for Calculating Young's Modulus From Simple Beam Test.

NOTE.—This nomograph can be used only for a specimen width of 1.00 in. and a distance of 2.00 in. between specimen supports. For information on use of nomograph see Section 5 (b).

### Calculations

5. (a) Calculate Young's modulus (in flexure) as follows:

$$E = \frac{Ll^3}{4wt^3(R_2 - R_1)}$$

where:

$E$  = Young's modulus (in flexure) in pounds per square inch,

$L$  = load in pounds added in weight pan,

$l$  = distance between supports in inches,

$w$  = width of specimen in inches,

$t$  = thickness of specimen in inches, and

$R_1$  and  $R_2$  = dial indicator readings.

(b) *Use of Nomograph.*—Calculation of Young's modulus from simple beam bending data is rather laborious even though two of the variables (width of test specimen and distance between specimen supports) are fixed in a given experimental apparatus. The nomograph in Fig. 4 has been constructed to simplify this procedure. To calculate Young's modulus by means of this nomograph, locate (1) the specimen thickness on the extreme right-hand scale, and (2) the observed deflection on the extreme left-hand scale. Connect these two points with a straight edge and locate the point of intersection of this straight edge and the center  $\frac{E}{L}$  scale (Note 1). Rotate the straight edge about this point on the  $\frac{E}{L}$  scale until it intersects the load scale at the appropriate point. At the intersection of the straight edge with the  $E$  scale read Young's modulus in pounds per square inch (Note 2). The nomograph in Fig. 4 can be used only for a specimen width of 1.00 in. and a distance of 2.00 in. between specimen supports.

NOTE 1.—The center scale represents the value of  $\frac{E}{L}$ . Since it is not necessary to know

the numerical value of this quantity, the scale has been left undivided.

NOTE 2: *Example.*—Calculate  $E$  for a sample 0.250 in. in thickness which gave a deflection of 0.018 in. under a load of 1.1 lb. Join 0.250 on the extreme right-hand scale and 0.018 on the extreme left-hand scale with a straight edge. Locate the point where this line crosses the center scale. Pivot the straight edge about this point till it is in alignment with the point 1.1 in the second scale from the left. Read 7800 psi. on the fourth scale from the left.

### Report

6. The curve for Young's modulus *versus* the temperature as the cold stiffening characteristic of each specimen tested shall be plotted. However, one or more limiting modulus values corresponding to predetermined temperatures, relative to the application of a particular product, shall be considered satisfactory for materials specifications, provided crystallization or other time-stiffening effects are known to be absent.

### Procedure (For detection of crystallization or other time effects.)

7. (a) The dimensions of the test specimens shall be measured and recorded and the specimens carefully centered on the saddles. With the refrigerator controls set at  $-4$  F. (Note 1), the air circulating fan shall be started. The temperature of the testing medium shall be maintained at  $-4$  F. for 15 min. before proceeding with the test.

NOTE 1.—The temperature of maximum rate of crystallization for natural rubber is approximately  $-4$  F. When testing neoprene GN for crystallization, a temperature of  $+32$  F. is preferable.

(b) The specimens shall be tested in accordance with the procedure described in Section 4 (b).

(c) The testing procedure shall be repeated for all the specimens in the cooling chamber at  $-4$  F. (Note 2) after which the temperature shall be maintained at  $-4$  F. for 72 hr.

NOTE 2.—If necessary, or desirable, the specimens being studied for crystallization or other time effects may be removed from the turntable saddle supports after the first set of measurements and may be maintained at  $-4^{\circ}\text{F}$ . for 72 hr. in an auxiliary cooling chamber. The second set of measurements may then be made by quickly transferring the specimens back to the saddle supports and proceeding as outlined above. This procedure, however, will result in considerable frosting of the interior of the refrigerator and should probably not be used for best results.

(d) The testing procedure described in Section 4 (b) shall then be repeated on all the specimens as described in Paragraph (b) after conditioning at  $-4^{\circ}\text{F}$ . for 72 hr.

## Calculations

8. From the data obtained both before and after conditioning for 72 hr., calculate Young's modulus from the formula given in Section 5.

## Report

9. If the calculated modulus after the 72-hr. conditioning period is considerably larger than before the 72-hr. exposure, the presence of crystallization or other time effects is indicated and shall be reported. Further evaluation of the magnitude or rate of crystallization or time-stiffening shall be governed by the expected service specification for a given material.

## EXPLANATORY NOTE

NOTE.—There are two types of change<sup>4</sup> that take place as flexible materials are cooled: (1) hardening without crystallization (second order transition or vitrification), (2) crystallization or other time effects (for example, hardening due to plasticizer incompatibility at low temperatures). The first change is purely thermal and is complete shortly after thermal equilibrium is reached. It is apparent as an immediate increase in the modulus, hardness, and brittleness. The second change depends on crystallization and progresses with time for rather long periods. This crystallization may take

place in the polymer itself or in some plasticizers or softeners. There is a temperature at which there is an optimum rate of crystallization. Above this temperature the tendency to crystallize is less and at lower temperatures the increased internal viscosity hinders orientation. Hence this change is most evident as an increase in modulus and hardness with time of conditioning at moderately low temperatures.

These considerations show that the time of conditioning necessary to measure the first type of change (second order transition or vitrification) is that necessary to insure thermal equilibrium. The second type of change (crystallization) can best be measured as a change in the hardness or modulus as function of time after the attainment of thermal equilibrium. The occurrence of crystallization or other time effects can be shown by measuring hardness or modulus at  $-4^{\circ}\text{F}$ . (see Note 1, Section 7) immediately after thermal equilibrium is reached and again after conditioning for 72 hr. at  $-4^{\circ}\text{F}$ . A significant increase in hardness or modulus after 72 hr. indicates crystallization or other time effects. Further evaluation of the magnitude or rate of crystallization or time-stiffening shall be governed by the expected service specification for a given material.

<sup>4</sup> Further discussion of these changes may be found in the following references:

Norman Bekkedahl, "Forms of Rubber as Indicated by Temperature-Volume Relationships," *Journal of Research*, Nat. Bureau Standards, Vol. 13, No. 3, September, 1934, pp. 411-431 (*Research Paper No. 717*).

Norman Bekkedahl and L. A. Wood, "Influence of the Temperature of Crystallization on the Melting of Crystalline Rubber," *Journal of Chemical Physics*, Vol. 9, No. 2, February, 1941, p. 193.

Norman Bekkedahl and L. A. Wood, "Crystallization of Vulcanized Rubber," *Industrial and Engineering Chemistry*, Vol. 33, March, 1941, p. 381.

Norman Bekkedahl and Russell B. Scott, "Specific Heat of the Synthetic Rubber Hycar O. R. from  $15^{\circ}$  to  $340^{\circ}\text{K}$ ," *Journal of Research*, Nat. Bureau Standards, Vol. 29, No. 1, July, 1942, pp. 87-95 (*Research Paper No. 1487*).

J. W. Liska, "Effect of Low Temperatures on Young's Modulus of Elastomers," *Industrial and Engineering Chemistry*, Vol. 36, p. 40 (1944).

R. F. Boyer and R. S. Spencer, *Journal of Applied Physics*, Vol. 15, p. 398, 1944.

# Standard Specifications for COTTON RUBBER-LINED FIRE HOSE FOR PUBLIC AND PRIVATE FIRE DEPARTMENT USE<sup>1</sup>



A.S.T.M. Designation: D 296 - 38

ADOPTED, 1934; REVISED, 1935, 1938.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 296; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These specifications cover 1½, 2, 2½, 3, and 3½-in. single-, double-, or triple-jacketed cotton, rubber-lined fire hose suitable for use in public or private fire departments.

## Cotton Fabric

2. (a) The jackets shall be well, evenly, and firmly made from good cotton, as free from unsightly defects, dirt, knots, lumps, and irregularities of twist as is consistent with good manufacturing practice.

(b) Each jacket shall be seamless and shall have the fillers woven around the hose throughout its length and the warps interwoven with and covering the fillers. The jackets may be separate or interwoven.

## Rubber Lining

3. (a) The lining shall be made of a properly vulcanized rubber compound

which will comply with all the tests specified in Sections 5 to 8, inclusive. It shall consist of not less than three calendered sheets in one solid body and shall be lap-jointed with the lap as small and as neat as is consistent with good results.

(b) The lining shall be smooth and practically free from pitting and other imperfections and from corrugations.

(c) The lining shall be of uniform thickness and within the following limits of thickness, exclusive of backing:

Internal Diameter of Hose, in.	Jackets	Lining Thickness, in.	
		Minimum	Maximum
1½ and 2	Single, and double.....	0.049	0.065
2½	Single, double and triple...	0.058	0.072
3	Double, and triple.....	0.058	0.072
3½	Double, and triple.....	0.072	0.095

(d) *Backing*.—The backing, if used, shall not exceed 0.028-in. in thickness, as measured from the bottom of the corrugations. It need not be of the same composition as the rubber lining, but the adhesion between the backing

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1928 to 1934, being revised in 1931.

and the cotton jacket shall conform to the requirements specified in Section 8.

### Chemical Composition

4. The rubber lining shall on chemical analysis conform to the following requirements as to chemical composition; all percentages shall be calculated on the weight of the total compound:

Organic acetone extract, max., per cent.....	4
Alcoholic-potash extract, max., per cent.....	1
Chloroform extract, max., per cent....	2.25
Ash (including carbon black), per cent.	50 to 57
Total sulfur (excluding barytes), max., per cent.....	3
Free sulfur, max., per cent.....	1.25

### Tensile Properties at 60 Days

5. (a) The tensile strength of the rubber lining shall not be less than 1600 psi. when tested within 60 days of the date of final vulcanization.

(b) *Elongation at Rupture*.—The elongation when rupture occurs in making the tensile strength test specified in Paragraph (a) shall be such that the original 2-in. gage length shall stretch to not less than 12 in.

(c) *Permanent Elongation*.—The permanent elongation or set, for tests made within 60 days, of samples cut transversely following a stretch from 2 to 10 in. shall not exceed 25 per cent and shall not exceed 30 per cent for samples cut longitudinally.

### Tensile Properties at 1 Year

6. (a) The tensile strength of the rubber lining shall not be less than 1200 psi. when tested after 60 days and within 1 yr. of the date of final vulcanization.

(b) *Elongation at Rupture*.—The elongation when rupture occurs in making the tensile strength test specified in Paragraph (a) shall be such that the

original 2-in. gage length shall stretch to not less than 10 in.

(c) *Permanent Elongation*.—The permanent elongation or set, for tests made within 1 yr., of samples cut transversely following a stretch from 2 to 8 in. shall not exceed 25 per cent and shall not exceed 30 per cent for samples cut longitudinally.

### Life Test

7. After subjection to a dry heat of  $158 \pm 2$  F. ( $70 \pm 1.1$  C.) for a period of four successive days of 24 hr. each and then placed in air at room temperature for 24 hr., the tensile strength of the rubber lining shall not be less than 900 psi.

### Friction Test

8. The adhesion between the lining and the cotton jackets shall be such that the rate of separation of a  $1\frac{1}{2}$ -in. strip of lining from the jackets shall not be greater than 1 in. per min. under a weight of 12 lb.

### Hydrostatic Pressure Tests

9. (a) Each length of single-jacketed hose shall be subjected to a hydrostatic pressure of 300 psi. for 5 sec. without leaking in the hose or at the couplings or breaking any threads in the jackets. Each length of double-, or triple-jacketed hose shall be subjected to a hydrostatic pressure of 400 psi. for 5 sec. without leaking in the hose or at the couplings, or breaking any threads in the jackets.

(b) *Elongation*.—The elongation of the hose when tested as specified in Paragraph (a), shall not exceed 10 per cent of the length for single-jacketed hose, and 8 per cent for double- or triple-jacketed hose, measured from an initial pressure of 10 psi.

(c) *Twist*.—The hose, when tested as specified in Paragraph (a), shall not

twist more than shown in the following table:

Internal Diameter of Hose, in.	Jackets	Twist, deg. per ft.
$1\frac{1}{2}$ and 2	Single.....	50
$2\frac{1}{2}$	Single.....	25
$1\frac{1}{2}$ and 2	Double.....	29
$2\frac{1}{2}$ , 3 and $3\frac{1}{2}$	Double, and triple.....	12.5

The twist requirements refer to the final twist, in a direction to tighten, rather than loosen the couplings. No final twist in a direction tending to loosen the coupling shall be permitted.

(d) *Warp and Rise*.—The hose, when tested as specified in Paragraph (a), shall not warp more than 20 in. from a straight line drawn from center to center of the couplings. Single-jacketed hose shall not rise from the level of the test table more than indicated in the following table, and no rise shall be permitted for double and triple-jacketed hose:

Internal Diameter of Hose, in.	Jackets	Permissible Rise, in.
$1\frac{1}{2}$ and 2	Single.....	7
$2\frac{1}{2}$	Single.....	4

### Bursting Strength Tests

10. A 3-ft. sample in every lot of 3000 ft., or fraction thereof, of hose, shall stand without failure, while lying either straight or curved to a radius of 27 in., a hydrostatic pressure as indicated in the following table:

Internal Diameter of Hose, in.	Jackets	Required Pressure, psi.
2 and $2\frac{1}{2}$	Single.....	500
$1\frac{1}{2}$ , 2 and $2\frac{1}{2}$	Double, and triple.....	600
3 and $3\frac{1}{2}$	Double, and triple.....	600

### Kink Tests

11. One full length in every lot of 500 ft., or fraction thereof, of hose shall stand without failure, while kinked, a hydrostatic pressure as indicated in the following table:

Internal Diameter of Hose, in.	Jackets	Required Pressure, psi.
$1\frac{1}{2}$ , 2 and $2\frac{1}{2}$	Single.....	300
$1\frac{1}{2}$ and 2	Double.....	350
$2\frac{1}{2}$	Double, and triple.....	350
3 and $3\frac{1}{2}$	Double, and triple.....	250

### Place of Hydrostatic Tests

12. All hydrostatic tests (Sections 9 to 11, inclusive) shall be performed either at the factory or at the destination, at the discretion of the purchaser. Arrangements for such tests shall be subject to arrangement reached between the manufacturer and the purchaser, at the time the order is placed.

### Diameter

13. The hose shall have an internal diameter as shown by a tapered plug gage of not less than  $1\frac{1}{2}$ , 2, 3, or  $3\frac{1}{2}$  in., as ordered. In the case of  $2\frac{1}{2}$ -in. hose, the internal diameter as measured in this manner shall not be less than  $2\frac{9}{16}$  in.

### Length

14. The hose shall be furnished in nominal lengths of 50 ft.

### Flexibility

15. The hose shall be flexible and easily coiled.

### Workmanship

16. The character of the workmanship shall be such as is incident to good manufacturing practice.

### Marking

17. Beginning at a point not less than 4 ft. from each end, each 50-ft. length of hose shall be stenciled at two places in indelible letters at least 1 in. in height with the name of the manufacturer and the month and year of manufacture.

### Sampling

18. (a) A sample not less than 8 in. in length shall be taken from each lot of

1000 ft., or fraction thereof, of hose for the tests specified in Sections 5 to 8, inclusive. All test specimens should be cut transversely from the sample, whenever possible.

(b) A sample not less than 4 in. in length shall be taken from each lot of 3000 ft., or fraction thereof, of hose for the chemical analysis specified in Section 4.

(c) Frequency of sampling for hydrostatic tests shall follow the requirements given in Sections 9 to 11, inclusive.

#### Methods of Testing

19. All measurements and tests necessary to determine compliance with the

specified requirements shall be made in accordance with the following method of the American Society for Testing Materials, except as otherwise prescribed in these specifications:

(a) *Chemical Analysis*.—Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297).<sup>3</sup>

(b) *Physical Tests*.—Tentative Methods of Testing Rubber Hose (A.S.T.M. Designation: D 380).<sup>3</sup>

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<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



# Standard Methods of TESTING FLAT RUBBER BELTING<sup>1</sup>



A.S.T.M. Designation: D 378 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 378; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. These methods cover the test procedure for evaluating the physical properties of the flat type of rubber belting which contains cotton duck reinforcement. The term "rubber" as used in these methods includes synthetic compounds as well as compounds of natural rubber. Such belting includes the types used for transmission of power or conveying and elevating material, and is classified as follows:

*Friction Surface Belt.*—Possessing no distinct rubber cover other than that resulting from the rubber frictioning process.

*Rubber Covered Belt.*—Possessing a distinct rubber cover extending over the outside and around the edges of the belt.

*Fabric Surface Belt.*—Possessing at least one face of plain uncoated duck.

## General Methods

2. (a) Except as otherwise specified in these methods for rubber belting, the following methods of test of the American Society for Testing Materials applicable in general to vulcanized rubber shall be complied with as required and are hereby made a part of these methods:

*General Physical Test Requirements.*

—Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15).<sup>3</sup>

*Tension Test.*—Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412).<sup>3</sup>

*Adhesion Test.*—Standard Methods of Test for Adhesion of Vulcanized Rubber (Friction Test) (A.S.T.M. Designation: D 413).<sup>3</sup>

(b) In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular belt, the latter shall take precedence.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to their present adoption as standard, these methods were published as tentative from 1933 to 1936. They were adopted in 1936, published as standard from 1936 to 1938, but withdrawn and republished as tentative from 1938 to 1941, being revised in 1939 and 1940.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Sampling

3. (a) Samples selected for purpose of tests shall be the full width and thickness of the belt and shall be of sufficient length to perform the desired tests. In general, the amount of sample required shall be as shown in Paragraph (b). The frequency of sampling for each size and type shall be one sample from each lot of 2000 ft. of belt or fraction thereof.

(b) The following lengths of samples are recommended:

Width of Belt, in.	Length of Sample when Tension Test is Required, in.	Length of Sample when Tension Test is not Required, in.
Under 8.....	32	12
8 to 18.....	26	8
Over 18.....	22	6

### Measurements of Dimensions

4. (a) *Width*.—The width shall be determined by laying across the belt a steel rule or tape graduated to sixteenths of an inch. The width shall be recorded in inches and fractions thereof to the nearest  $\frac{1}{16}$  in.

(b) *Length*.—In determining the length of a belt the measurement shall be made with a steel tape. The reference marks shall be parallel and in the same plane. In determining the length of an endless belt the measurement shall be made on the inside surface using three or more reference marks.

(c) *Thickness of Belts and Covers*.—The thickness of the belts and covers shall be determined by means of a micrometer graduated to thousandths of an inch, and having a presser foot  $0.25 \pm 0.01$  in. in diameter exerting a pressure of  $3 \pm 0.1$  oz. on the test specimen. The pressure shall be applied by means of a weight.

(d) *Thickness of Covers*.—The thickness of the top or bottom cover shall be the minimum thickness found on the test specimen prepared for the adhesion test and shall be determined as follows:

The over-all thickness of the test specimen shall first be measured. The rubber cover shall then be stripped from one surface and the thickness of the carcass, including the remaining cover, shall be measured. The difference between the first and second measurements will represent the thickness of the cover removed. The same procedure shall be followed on the test specimen from which the first cover has been removed, when the thickness of the other cover is required. When determining the cover thickness of a belt it is essential to have the specimen lie flat on the platform of the micrometer and the projecting ends must be properly supported to prevent tilting. The test specimen may consist of a small section cut from the belt.

(e) *Thickness of Cover when Tie Cloth is Used*.—The thickness of the cover shall include the tie cloth.

### Test Conditions

5. Tests shall be made within a temperature range of 70 to 90 F. (21 to 32 C.). The samples shall be kept at a temperature between these limits for at least 30 min. previous to the time of testing. The values obtained in determining the physical properties under these methods will differ, depending upon the age of the samples; therefore, the test shall be made within a definite time limit which shall not exceed 60 days from receipt of the material by the purchaser.

### Number of Tests

6. (a) When minimum requirements are established, one specimen shall be tested for each physical characteristic required in the detailed specifications for a particular belt. If the results do not meet the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified require-

ments, the material represented by the sample shall be considered to have failed to meet the specifications.

(b) If minimum requirements are not established, at least three specimens shall be tested and the results averaged.

## TENSION TESTS OF RUBBER PARTS

### Apparatus

7. (a) *Testing Machine.*—Tensile strength and elongation tests shall be made on apparatus accurate to within plus or minus 1 per cent and provided

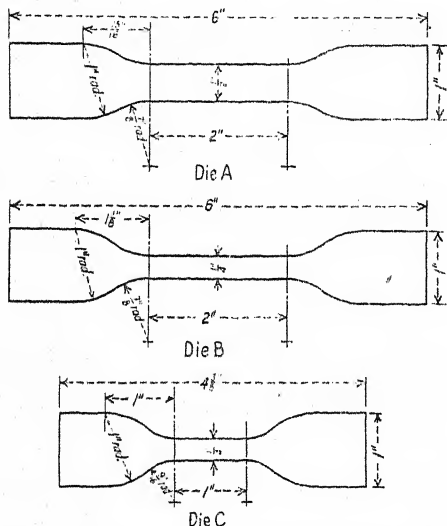


FIG. 1.—Tensile Strength and Elongation Test Specimens.

with an accurate device for indicating the actual maximum load at rupture. The preferred type of apparatus is a power-driven machine of the inclination balance or pendulum type, but a spring balance, accurately calibrated, may be used.

(b) *Grips for Testing.*—The grips for holding the test specimen shall be such that they tighten automatically, exerting a uniform pressure, proportional to the applied tension, across the full width of the enlarged end of the test specimen.

The moving grip shall move at a constant speed of 20 in. per min.

### Test Specimens

8. (a) Tests for tensile strength and ultimate elongation shall be made only on rubber covers from which standard dumbbell-shaped specimens at least  $\frac{3}{64}$  in. in thickness can be prepared. The test specimens shall be stamped from the covers in a transverse direction, at right angles to the axis of the belt.

(b) The rubber parts to be tested shall be separated from the rest of the belt without the use of gasoline, if possible. If it is necessary to employ gasoline to effect the separation, it shall be straight-run gasoline of the quality known as aviation grade and shall have the characteristics listed in the following table:

Specific gravity at 60/60 F.. 0.685 to 0.705  
Distillation limits:

Initial boiling point.....	100 to 120 F.
10 per cent evaporated.....	128 to 143 F.
20 per cent evaporated.....	140 to 155 F.
30 per cent evaporated.....	150 to 164 F.
40 per cent evaporated.....	156 to 174 F.
50 per cent evaporated.....	164 to 184 F.
60 per cent evaporated.....	173 to 194 F.
70 per cent evaporated.....	182 to 207 F.
80 per cent evaporated.....	193 to 223 F.
90 per cent evaporated.....	209 to 245 F.
95 per cent evaporated.....	224 to 260 F.
Dry.....	250 to 286 F.
Recovery, min. ....	97 per cent

The gasoline shall contain no lead compounds and shall not leave an appreciable amount of oily residue when evaporated.

After use of gasoline, the test specimen shall be allowed to rest, with frequent turning to permit free evaporation from all parts of its surface, at least 1 hr. before being tested.

(c) *Preparation of Test Specimen.*—The test specimen shall be stamped out from the separated cover with a steel die conforming to Fig. 1, A, B, or C. Die B shall be used when the size of the sample and the tension and stretch limits of the testing machine permit, with exceptions as explained below. If the

tension limit is too low for the machine, die A shall be used. If the stretch is too high for the machine, die C shall be used. Die C shall also be used for samples which are too small for die A or B. The dies shall be kept sharp and free from nicks to avoid leaving ragged edges on the rubber. Cutting may be facilitated by wetting the rubber surface and cutting edges of the die. The rubber shall be rested on a smooth, slightly yielding surface that will not injure the blade. Care shall be taken that the cut edges are perpendicular to the other surfaces of the specimen and have a minimum of concavity.

(d) *Buffing*.—The specimen shall be buffed only to the extent necessary to remove surface corrugations, irregularities, or excess thickness and to insure smooth faces. Buffing shall be done without excessive heating and the test specimen shall be allowed to rest after buffing for at least 10 min. before being tested.

(e) *Marking*.—Parallel gage lines 1 to 2 in. apart, depending upon the die used, shall be marked on the test specimen at right angles to the longitudinal axis by means of an accurate stamp or bench marker. Care shall be taken to make fine lines and to avoid injuring the specimen.

(f) *Measurement of Test Specimen*.—The thickness of the test specimen shall be the minimum thickness found between the marks on the constricted portion, using the stand micrometer described in Section 4 (c). The minimum thickness and width between the gage marks shall be taken in calculating the area to be used in determining the tensile strength of the specimen.

(g) *Number of Test Specimens*.—Three specimens per sample shall be tested for determinations of tensile

strength, elongation, and stress-strain properties, except as noted in Section 6 (a). Results on specimens which break outside of the straight reduced section or which are found to be imperfect shall be discarded and retests shall be made. In case the ultimate tensile strengths fail to check within 10 per cent of the highest value obtained, additional specimens shall be tested until two or more such check results are obtained. The final values shall be the averages of the check tests, discarding the determinations on specimens which are outside of the above limits.

#### Procedure

9. (a) The test specimen shall be placed in the jaws. The jaws shall be separated at the specified rate of speed until rupture takes place. The distance between the centers of the gage marks noted at the instant of rupture shall be measured, using a rule or tape graduated to eighths of an inch or smaller divisions.

(b) *Tensile Strength*.—The tensile strength in pounds per square inch shall be calculated from the breaking load indicated by the machine and the original cross-sectional dimensions of the test specimen (Section 8 (f)).

(c) *Elongation*.—The elongation shall be the difference between the original gage length and the total distance between the gage marks at the time of rupture as measured in Paragraph (a) and shall be expressed as a percentage of the original gage length.

#### OIL IMMERSION TESTS

##### Purpose

10. These oil immersion tests are intended for use only on rubber covers of belts that are being evaluated for service in which they come into contact with oils as, for example, conveyor belts handling oiled, dustless coal.

### Apparatus

11. The immersion apparatus shall consist of a glass container fitted tightly with a cork stopper, and an efficient reflux condenser. A glass test tube having an outside diameter of 38 mm. and an over-all length of 300 mm. may be employed. Use of a thermostatically controlled liquid bath is recommended for heating the apparatus.

### Solvent

12. The solvent shall be a neutral, pale, paraffin-base oil. The oil shall be yellow in color with blue fluorescence, free from turbidity, and shall conform to the following requirements:

Test	A.S.T.M. Designation <sup>a</sup>	Minimum	Maximum
Specific gravity, per cent ...		0.878	0.882
Flash point, deg. Fahr. ...	D 92	330	340
Saybolt Universal Viscosity at 122 F., sec. ....	D 88	61	66

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials: Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92),<sup>4</sup> and Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>4</sup>

### Test Specimens

13. (a) Tests for tensile strength, ultimate elongation, and increase in volume (swelling) shall be made only on the rubber covers from which standard dumbbell test specimens at least  $\frac{3}{16}$  in. in thickness can be prepared. Test specimens shall be prepared in accordance with the procedure described in Section 8.

(b) For the determination of changes in volume, rectangular test specimens having dimensions 1 by 2 by 0.075 in. shall be used.

### Changes in Tensile Strength and Elongation

14. Three specimens shall be tested for tensile strength and elongation in accordance with the procedure described

in Section 9, and the results averaged to give the properties of the rubber before immersion. Three additional specimens shall be placed in the immersion apparatus and completely surrounded by the solvent. With the reflux condenser in place, the apparatus containing the specimens shall be maintained at a temperature of  $200 \pm 2$  F. ( $93 \pm 1$  C.), for a period of 48 hr. At the end of this period, the specimens shall be removed, dipped once in acetone at room temperature, and blotted lightly with filter paper. The specimens shall then be suspended or placed on a screen and allowed to dry at room temperature in air protected from drafts for 4 hr., after which the tensile strength and elongation after immersion shall be determined as described in Section 9, except that the measurements for cross-sectional area and test length between gage marks shall be taken after immersion and drying.

### Increase in Volume (Swelling)

15. The initial water displacement of the specimen shall be determined by weighing it in air to the nearest milligram ( $W_1$ ) and in distilled water at room temperature ( $W_2$ ), the difference being the weight of the water displaced by the specimen. The specimen shall then be dipped quickly into SDA-2B alcohol, blotted dry with filter paper, and placed in the immersion apparatus. Then 100 ml. of the solvent shall be added, and, with the reflux condenser in place, the apparatus containing the specimen shall be maintained at a temperature of  $200 \pm 2$  F. ( $93 \pm 1$  C.) for a period of 48 hr. At the end of the required length of immersion time, the specimen shall be removed and cooled to room temperature by being transferred to a cool, clean portion of the test liquid. It shall then be dipped quickly into SDA-2B alcohol

at room temperature, blotted lightly with filter paper, placed in a tared weighing bottle, and weighed ( $W_3$ ). It shall next be removed from the bottle and weighed in distilled water ( $W_4$ ), in immediate consecutive procedure, to determine the water displacement after test. It is important that all air bubbles clinging to the test specimens be removed during the weighing in distilled water. When data is desired on the progressive changes occurring with increasing time of immersion, the test specimen shall be immediately removed from the water after making this determination, dipped quickly into SDA-2B alcohol at room temperature, blotted lightly with filter paper to remove the water and alcohol, and replaced in the test liquid with the least possible lapse of time.

#### Calculations

16. (a) *Changes in Tensile Strength and Elongation.*—The deterioration in tensile strength and ultimate elongation as shown by the difference between the determinations before and after immersion shall be calculated as a percentage of the original property.

(b) *Increase in Volume.*—The increase in volume shall be calculated as follows:

Percentage increase in volume =

$$\frac{(W_3 - W_4) - (W_1 - W_2)}{(W_1 - W_2)} \times 100$$

where:

$W_1$  = initial weight of specimen in air,

$W_2$  = initial weight of specimen in water,

$W_3$  = weight of specimen in air after the immersion test, and

$W_4$  = weight of specimen in water after the immersion test.

#### ADHESION TESTS

##### Test Specimens

17. The adhesion test specimens shall be prepared by accurately cutting a strip 1 in. in width longitudinally of the test sample from belts under 8 in. in width, and transversely from belts 8 in. and over in width, using a sharp tool which will leave clean-cut edges. The test specimen shall be at least 6 in. in length. The various plies shall be separated at one end of the test specimen a sufficient distance to permit attaching the jaws of the testing clamp.

##### Procedure

18. (a) The adhesion between plies shall be determined by the dead weight method, that is, through the application of a specified or known weight on successive plies of a specimen 1 in. in width in such a manner as to cause separation. Adhesion shall be expressed numerically as the average rate of separation in inches per minute.

(b) The test specimen shall be suspended by the separated end, all plies of which except the one under test shall be clamped in the jaws of the testing clamp. A minimum weight sufficient to maintain the test specimen in approximately a vertical position shall be attached to its lower end. The first ply shall have the specified weight attached to the separated end with suitable provision for supporting and releasing it slowly without jerking. A mark shall be placed on the line of separation at the beginning and at the end of the test. After the weight is removed at the end of the test, the distance between the marks shall be measured. The rate of separation shall be determined by observing the duration of the test with a stop watch or a watch having a second hand, and by measuring, after the weight has been

removed, the length stripped between the marks placed at the beginning and end of the test on the portion other than that stripped. Under these conditions the rate of separation shall not exceed 1 in. per min.

(c) The test shall be continued on the successive plies by releasing the separated end of each from the stationary clamp and reattaching the specified weight. This procedure shall be followed until two plies only remain within the grip of the stationary clamp. No tests shall be made between the last two plies of the test specimen (Note). When tests are desired on these plies, a second specimen shall be prepared and shall be tested, starting from the opposite side in accordance with the procedure described in Paragraphs (a) and (b).

NOTE: The last two plies are omitted from the test because sufficient rigidity cannot be maintained at the point of separation to obtain the approximate condition of separation in a vertical plane.

(d) The adhesion between the rubber covers and the body of the belt shall be determined on the test specimen prepared for ply adhesion except that no tests for cover adhesion shall be made on rubber covers less than  $\frac{3}{16}$  in. in thickness. The procedure shall be the same as that described for ply adhesion in Paragraphs (a) and (b).

(e) On rubber-covered belting constructed with a tie cloth or breaker fabric between the outside ply and cover, the adhesion between the body of the belt and covering including the tie cloth, or between the tie cloth and adjacent rubber cover shall be determined on separate test specimens in accordance with the procedure described in Paragraph (b).

## BEADING TEST<sup>5</sup>

### Procedure

19. The beading in all friction-surface belting 4 in. in width or over shall be tested as follows: A 4-in. transverse section of belting shall be taken and beginning on the seamless side, the plies shall be removed until three plies only remain intact. A line shall be drawn down the middle of the beading and then on each side of this line, and  $\frac{5}{8}$  in. distant from it, two parallel lines shall be scribed. The section shall be inserted in a vise so that the jaws grip the specimen coincident with the two outer lines. The vise shall be tightened until the inner surfaces of the doubled specimens just touch at the top of the vise, and the specimen shall be held in this position for 10 min. Under this test the beading shall not crack or loosen in the seam.

## TENSION TESTS OF BELTING

### Apparatus

20. The specimens shall be tested in a suitable vertical testing machine equipped with wedge-shaped jaws. The base of the jaws shall be at right angles to the longitudinal center of the test specimen when correctly placed in the jaws. The base surfaces of the two edges of each set of jaws shall be in the same horizontal plane and adequate means shall be provided to lock the two halves of the jaws so that they will travel together in parallelism. Means shall be provided to hold the jaws in convenient position for correctly inserting the test specimen as well as to loosen them after each test. The heads of the testing machine shall be constructed to provide rigid support for

<sup>5</sup> The beading is considered by many rubber technologists as having no relation to the service value of belting, except for some specialized requirement. The beading test is, therefore, not recommended for general use.



the jaws. The inner surfaces of the wedge-shaped slots shall be machined to within 0.005 in. of a true plane. The included angle of the two sides shall be from 17.5 to 22 deg. and this angle must be identical with that of the jaws.

### Test Specimens

21. For belting up to 5 in. in width, inclusive, a longitudinal tension test specimen 20 in. in length and the full width of the belt shall be cut from the sample. For belting between 5 and 6 in. in width,  $\frac{1}{2}$  in. shall be removed from each edge. For belting 6 in. and over in width the test specimen shall be 20 in. in length and 5 in. in width, and the edges of the specimen shall be cut parallel to and at least  $\frac{1}{2}$  in. from the edges of the belt. The thickness of the test specimen shall not exceed 4 plies. Excess plies shall be removed by stripping, removing them alternately from each face. When testing rubber-covered belts, rubber covers  $\frac{3}{4}$  in. and over in thickness shall be stripped from the test specimen.

### Procedure

22. (a) *Tensile Strength*.—The grooves and gripping surfaces of the jaws shall be cleaned and the edge surfaces and backs shall be well lubricated and kept free of any foreign matter. Slippage may be prevented by the use of coarse emery cloth which shall be inserted between the corrugations of the jaws and the test specimen, with the rough side toward the specimen, and this cloth shall be renewed frequently. Great care must be used to prevent the loose particles of grit which fall from the emery cloth from lodging upon any of the pressure surfaces during the test. The test specimen shall be tested in the machine described in Section 20. Care shall be taken to place the specimen squarely in the jaws of the machine.

The jaws shall be 12 in. apart when the test specimen is inserted and the rate of separation shall be between  $\frac{3}{4}$  and 2 in. per min., inclusive. Test specimens that break in the jaws or that, upon examination, are found to be imperfect shall be discarded, and retests shall be made unless the results obtained in the first test exceed minimum values that may be required by detailed specifications. From the breaking strength thus determined, the tensile strength of the belt per inch of width per ply shall be calculated and reported.

(b) *Elongation*.—The elongation, when required, shall be taken during the performance of the tensile strength test. For this purpose, parallel reference lines 8 in. apart shall be marked across the middle of the test specimen at right angles to the longitudinal axis. The elongation shall be determined by means of a steel scale or suitable extensometer. When an extensometer is used, it shall be pinned or held on the specimen with the points on the 8-in. reference lines and the scale set at zero. The load shall then be applied at the specified rate of speed and the elongation at the breaking point shall be noted.

(c) *Stress-Strain*.—If the stress-strain characteristics of the belt are desired, they may be obtained simultaneously with the tensile strength determination. In this case, elongation readings of the steel scale or extensometer shall be taken at intermediate points. The total load on the belt and the percentage elongation for each increment increase in load per inch per ply shall be reported as well as the total load and percentage elongation at failure.

### INSPECTION AND REJECTION

#### Inspection

23. (a) All tests and inspection shall be made at the place of manufacture



prior to shipment, unless otherwise specified. The manufacturer shall afford the inspector, without charge, all reasonable facilities to test and inspect the belting.

(b) The purchaser may make the tests and inspection to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser, not later than 60 days after receipt of the material.

#### **Retests and Rejection**

24. (a) Any material which fails in one or more tests may be resampled and retested at the expense of the manufacturer. For this purpose two additional samples shall be selected from the belt for the test which failed to meet the requirements. Failure of either of the retested samples shall be cause for final rejection.

(b) Rejected material shall be disposed of as directed by the manufacturer and at his expense.

# Standard Specifications for

## ELECTRICAL GLOVES<sup>1</sup>

VOLTAGE RATING OF GLOVES, 3000 VOLTS<sup>2</sup>



A.S.T.M. Designation D 120-40

ADOPTED, 1940.<sup>3</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 120; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These specifications cover electrical workers' rubber gloves of two classes, as follows:

*Class A.*—Gloves intended for use without external protection.

*Class B.*—Gloves intended for use with external protection of leather or other material.

### Manufacture

2. The gloves shall be made by the "dipping" or other seamless process, of high grade rubber or rubber compound properly vulcanized. Unless otherwise specified, cuff edges shall be finished with a roll or a reinforcing strip of rubber.

### Electrical Properties

3. (a) *Voltage and Leakage Tests.*—Each glove shall withstand an alternat-

ing potential of 10,000 v. (r.m.s. value) for 3 min. At 60 cycles the leakage current shall not exceed 10 ma. (total current) at any time during the test. If tested at any other frequency ( $x$  cycles) the permissible current leakage ( $I_x$ ) may be determined by the following formula:

$$I_x = 10 \frac{x \text{ cycles}}{60 \text{ cycles}}$$

(b) *Dielectric Strength.*—Each glove shall be capable of withstanding the application of the following voltage (r.m.s. value) for 1 min., at 60 cycles, without showing a leakage current in excess of that given in the following:

	Volts	Leakage Current, max., Milliamperes
Class A. ....	16 000	18
Class B. ....	14 000	14

### Physical Properties, as Received

4. A test specimen cut from any glove shall conform to the following requirements as to physical properties:

(a) *Tensile Strength.*—The tensile strength shall be not less than 1800 psi.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Voltage stress through wall of glove limited to 3000 v.

<sup>3</sup> Prior to adoption as standard, these specifications were published as tentative from 1921 to 1923, being revised in 1922. They were adopted in 1923, published as standard from 1923 to 1937, but withdrawn and republished as tentative from 1937 to 1940. Title editorially revised in 1941.

(b) *Elongation*.—The total elongation in 1 in. at rupture shall be not less than 600 per cent (1 in. stretched to 7 in.).

(c) *Set*.—The set in 1 in. following an elongation of 500 per cent (1 in. stretched to 6 in.) shall not exceed 0.25 in.

### Physical Properties, After Aging

5. (a) After being subjected to a temperature of  $158 \pm 2$  F. ( $70 \pm 1$  C.) in circulating air for 7 days, the tensile strength and elongation shall be not less than 80 per cent of the original.

(b) After being subjected to a temperature of  $158 \pm 2$  F. ( $70 \pm 1$  C.) in oxygen at a pressure of 300 psi. for 4 days, the tensile strength and elongation shall be not less than 80 per cent of the original.

### Size and Length

6. (a) *Size*.—Size shall be expressed in inches as measured around the palm at the base of the knuckles. The permissible variation in size shall be plus or minus  $\frac{1}{4}$  in.

(b) *Length*.—Length shall be expressed in inches as measured from the tip of the second finger to the edge of the cuff. Unless otherwise specified, the length shall be 14 in. The permissible variation in length shall be plus or minus  $\frac{1}{2}$  in.

### Thickness

7. The respective thicknesses of the two classes of gloves in inches shall be within the following limits:

	Part of Glove other than Crotch		Crotch	
	Maxi- mum	Mini- mum	Maxi- mum	Mini- mum
Class A.....	0.065	0.050	0.065	0.038
Class B.....	0.050	0.035	0.050	0.026

### Workmanship and Finish

8. Gloves shall be free from patches,

blisters, pinholes, cracks, protuberances, imbedded foreign matter, or other physical defects which can be detected by thorough surface inspection.

### Marking

9. Unless otherwise specified each glove shall be marked with the name of the manufacturer and the size of glove. Such marking shall be confined to the back of the glove and shall be so done as not to injure the glove. Indelible and nonconducting ink shall be used.

### Packing

10. Each pair of gloves shall be packed in an individual stiff paper box of sufficient strength to properly protect the gloves. The end of the box shall be marked with the glove size and in the case of class B gloves with the words "For use with Protectors."

### Guarantee

11. The manufacturer shall replace, without charge to the purchaser, gloves which, at any time within a period of 8 months from the date of initial test, fail to pass the tests prescribed in these specifications; provided the gloves have been properly stored in their original boxes.

NOTE.—"Proper storage" is to be interpreted to mean that gloves are not to be stored directly above or in proximity to steam radiators or other sources of artificial heat. It is desirable that the ambient temperature shall not exceed 90 F. (32 C).

### Inspection

12. Inspection and test, unless otherwise specified, shall be made within two weeks of receipt of the material by the purchaser at his own expense at such place as he may designate.

### Rejection

13. (a) Each glove that fails to meet the requirements prescribed in Section

3 (a) and (b) or Section 8 shall be rejected. Rejection of gloves failing to meet the requirements of Section 6, 9, or 10 is optional with the purchaser.

(b) If any of the measurements of thickness made as prescribed in Section 16, or any additional measurements which the purchaser may elect to make, fall below the minimum thicknesses specified in Section 7, the glove shall be rejected. If any of the measurements exceed the maximum thicknesses speci-

20 fail to meet the requirements of Sections 4 and 5, the entire shipment shall be rejected.

### Marking Rejected Gloves

14. All rejected material shall be returned to the manufacturer without being defaced by rubber stamp or other permanent marking, except in gloves which have been punctured on electrical tests. These shall be stamped, punched, or cut to indicate that they are unfit for electrical use.

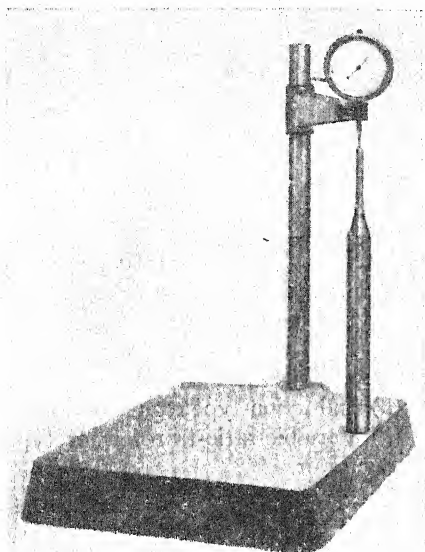


FIG. 1.—Dial Micrometer.

fied, rejection is optional with the purchaser.

(c) If rejections under Paragraphs (a) and (b) exceed 25 per cent of the shipment, the entire shipment may be rejected without further tests.

(d) If 25 per cent or more of the gloves subjected to the test prescribed in Section 3 (b) fail to pass the test, the entire shipment shall be rejected.

(e) If the average of the results for either the tensile strength, elongation, or set tests made as prescribed in Section

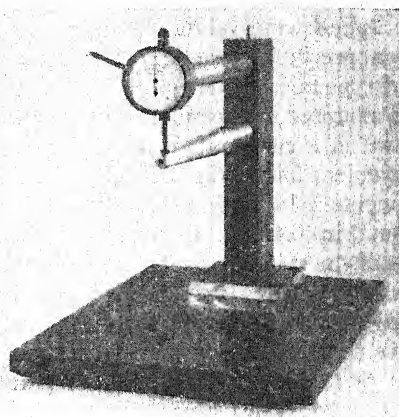


FIG. 2.—Dial Micrometer.

### METHODS OF TESTING

#### Order of Testing

15. The order of procedure in testing rubber gloves shall be preferably be as follows:<sup>4</sup>

(a) Inspection of inside and outside surfaces (Section 8),

(b) Measurement of length (Section 6 (b)),

(c) Measurement of size (Section 6 (a)),

(d) Measurement of thickness (Section 7),

(e) Voltage and leakage tests (Section 3 (a)),

<sup>4</sup> This procedure has been found by experience to minimize the expense of the inspection and testing particularly where an appreciable number of gloves fail to meet the requirements as to workmanship or dimensions.

- (f) Dielectric strength test (Section 3 (b)), and  
 (g) Physical properties (Section 4).

### Thickness Measurements

16. (a) *Apparatus*.—The thickness measurements shall be made with any micrometer graduated to mils or tenths of mils, having a ratchet friction or spring attachment. The tension on the friction attachment shall be so adjusted that, when the spindle has come to a stop, the specimen can be moved between the spindle and anvil without causing any appreciable stretching of the rubber. Dial-type micrometers graduated to mils and mounted in a manner similar to that shown in Figs. 1 and 2 are particularly convenient for making all of these measurements. In both cases the anvil is about  $\frac{1}{4}$  in. (6.4 mm.) in diameter and the foot of the spindle about  $\frac{3}{8}$  in. (3.2 mm.) in diameter.

(b) *Procedure*.—The thickness of each glove shall be measured at not less than four points on the face and four points on the back. Measurements shall be made at one or more points in the crotch of the thumb and index finger and in the crotches between the fingers.

### Electrical Tests

#### Voltage and Leakage Tests

17. (a) Each glove shall be tested for voltage and leakage current in fresh water at normal room temperature (60 to 90 F. (15 to 32 C.)) by immersing it in the water to within about  $1\frac{1}{2}$  to 2 in. of the edge of the cuff and filling the glove with water up to the same point. The water inside the glove and that outside the glove form the test electrodes. These shall be conveniently connected to the testing transformer by means of a chain suspended in the glove and by direct connection to the containing vessel.

(b) *Source of Test Voltage*.—The testing voltage shall be obtained from a testing equipment which shall be capable of delivering not less than 1 $\frac{3}{4}$  kva. for each glove tested but in no case shall the rating of the testing transformer be less than 2 kva. The frequency of the testing voltage shall be not more than 65 cycles.

(c) *Application of Test Voltage*.—The potential shall be applied at a low value and gradually and steadily raised at a rate of approximately 1000 v. per sec. until the prescribed testing voltage is reached. The test period shall be counted from the instant when the prescribed testing voltage is reached.

(d) *Voltage Regulation*.—The method of regulating the test voltage shall be such that the wave shape of the voltage produced does not have a deviation factor (Note) larger than 10 per cent. Acceptable methods of voltage regulation include the following:

- (1) Field regulation of the alternator supplying the transformer,
- (2) Induction-type regulator, and
- (3) Variable-ratio-transformer type of regulator.

NOTE.—The deviation factor of a wave<sup>5</sup> is the ratio of the maximum difference between corresponding ordinates of the wave and of the equivalent sine wave to the maximum ordinate of the equivalent sine wave when the waves are superposed in such a way as to make this maximum difference as small as possible.

(e) *Voltage Measurement*.—The testing voltage shall be measured by one of the following methods:

- (1) A calibrated potential transformer with a voltmeter.
- (2) Any properly calibrated commercial type of alternating-current voltmeter connected to the low-tension side of the transformer in conjunction with the ratio of transformation

<sup>5</sup> Standard No. 7, Alternators, Synchronous Motors and Synchronous Machines in General, Par. 7-650 Deviation Factor of Wave Defined, Am. Inst. Electrical Engrs., December, 1927.

of the transformer provided that the ratio is definitely known for all test conditions.

(f) *Leakage Current Measurement.*—

The leakage current shall be measured directly by inserting a milliammeter in series with each individual glove in turn.

NOTE.—It is customary to make high voltage tests of this character with one end of the circuit grounded, the containing vessel usually being the grounded end of the circuit. The milliammeter should therefore be connected in at the grounded end of the circuit and shunted by a short-circuiting automatic self-closing switch which will therefore always keep the circuit closed except at the instant of reading and thus maintain an uninterrupted ground.

### Dielectric Strength Tests

18. (a) Not less than ten per cent of the gloves which have passed all of the tests previously prescribed in these specifications shall be selected at random and tested for compliance with Section 3 (b).

(b) One per cent of the gloves (in no case less than two gloves) which have passed all the tests previously prescribed in these specifications shall be selected at random and tested for ultimate dielectric strength. The test shall be made in accordance with the general method described in Section 17.

The potential shall be raised steadily and gradually at a rate of approximately 1000 v. per sec. until failure occurs, the voltage at failure being recorded.

### Physical Tests

#### Test Specimens

19. Four or more test specimens shall be cut from each glove tested under Section 18 (b). The shape of these specimens shall be in substantial accord with that prescribed in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation D 412) of the

American Society for Testing Materials.<sup>6</sup> The essential dimensions of this specimen are as follows: Total length,  $4\frac{3}{8}$  in.; width, 1 in. with a reduced section at the center,  $\frac{1}{4}$  in. in width and about 1 in. in length, with a radius of curvature between the ends and the reduced section of  $\frac{9}{16}$  in.

### Tensile Strength, Elongation, and Set Tests

20. The tests for tensile strength, elongation, and set shall be made in accordance with the Standard Methods D 412.<sup>6</sup> The essential features of these methods are as follows:

(1) Temperature of testing room and of specimens, between 70 and 90 F.

(2) Rate of separation of jaws of testing machine, 20 in. per min.

(3) Tensile strength and elongation shall be made on one specimen, and set test on a separate specimen.

(4) Set test shall be made by stretching the 1-in. gage length to 6 in., holding for 10 min., and measuring the distance between the gage marks 10 min. after release.

### Accelerated Aging Tests

21. The tests for accelerated aging of the rubber compound shall be made in accordance with the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (A.S.T.M. Designation: D 572)<sup>6</sup> and the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573)<sup>6</sup> of the American Society for Testing Materials.

<sup>6</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

*Standard Specifications for*  
**RUBBER MATTING FOR USE AROUND ELECTRIC  
APPARATUS<sup>1</sup>**

VOLTAGE RATING OF MATTING, 3000 VOLTS



A.S.T.M. Designation: D 178 - 24

ADOPTED, 1924.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 178; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Scope**

1. These specifications cover rubber matting for use as a floor covering near electric apparatus.

**Manufacture**

2. The matting shall consist of a rubber compound corrugated on one surface and backed with cotton fabric, or having one or more cotton fabric inserts, the back of the matting being finished with cloth imprint.

**Electrical Properties**

3. (a) *Voltage Test*.—The entire area of the matting as nearly as practicable shall be tested between electrodes consisting of rectangular metal sheets having smoothly rounded edges and corners, of

any convenient length, and a width such that arcing around the edges of the matting will not occur, the tests being made progressively until the entire length of the matting has been covered. A potential of 15,000 v. shall be applied for 1 min. at each position of the electrodes. The matting shall not puncture, become appreciably warm at any spot, or show any other sign of weakness.

(b) *Dielectric Strength*.—The matting shall not fail at less than 40,000 v. when tested in air between 2-in. disk electrodes with edges rounded to a radius of  $\frac{1}{4}$  in., the voltage being applied to failure at the rate specified in Section 10 (b).

**Physical Properties**

4. (a) *Tensile Strength*.—The tensile strength shall be not less than 700 psi.

(b) *Elongation*.—The total elongation in 2 in. at rupture shall be not less than 250 per cent (2 in. stretched to 7 in.).

(c) *Set*.—The set in 2 in. following an

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1923 to 1924. Editorially revised and rearranged in 1939.

elongation of 150 per cent (2 in. stretched to 5 in.) shall not exceed 0.50 in.

### Thickness

5. The thickness, unless otherwise specified, shall be not less than  $\frac{1}{4}$  in. when measured over the corrugations and not less than  $\frac{1}{8}$  in. at the root of the corrugations.

### Workmanship and Finish

6. Matting shall be free from patches, blisters, pin-holes, cracks, protuberances, embedded foreign matter, or other physical defects which can be detected by thorough surface inspection.

### Inspection

7. The matting shall be inspected and tested within four weeks of the date of delivery.

### Rejection

8. (a) Any piece or roll of matting which fails to pass any of the requirements of these specifications shall be rejected.

(b) In case of failure to pass any one of the tests specified in Sections 3 (b) and 4, the piece or roll of matting represented by the sample subjected to these tests shall be rejected.

## SAMPLING AND METHODS OF TESTING

### Sampling

9. Not less than one sample shall be taken from each piece or roll of matting and subjected to the tests specified.

### Electrical Test

10. (a) *Apparatus*.—The test voltage shall be obtained from a testing equipment, no part of which has a capacity of less than  $\frac{1}{2}$  kva. per sq. ft. of electrode surface. In no case shall the rating of any part of the testing apparatus be

less than 5 kva. The frequency of the test voltage shall be not more than 65 cycles.

(b) *Application of Test Voltage*.—The potential shall be applied at a low value and gradually and steadily raised at a rate of approximately 800 to 1000 v. per sec. until the prescribed test voltage is reached. The test period shall be counted from the instant when the prescribed test voltage is reached.

(c) *Voltage Regulation*.—The method of regulation shall be one which does not distort the wave form of the test voltage from a sine wave. Acceptable methods include:

- (1) Field regulation of the alternator supplying the transformer,
- (2) Induction type regulator,
- (3) Variable-ratio-transformer type of regulator, and
- (4) Potentiometer type of rheostatic control where the current in the portion of the potentiometer resistance in parallel with the primary of the transformer is at least five times the exciting current of the transformer.

(d) *Voltage Measurement*.—The test voltage shall be measured by one of the following methods:

- (1) A calibrated potential transformer with a voltmeter,
- (2) Any properly calibrated commercial type of alternating-current voltmeter connected to the low tension side of the transformer, in conjunction with the ratio of transformation of the transformer, provided that the ratio is definitely known for all test conditions, or
- (3) A properly calibrated electrostatic voltmeter connected directly across the matting under test.

### Methods of Physical Testing

11. The tests for determining the physical properties specified shall be



made on the rubber exclusive of fabric and in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation:

D 412) of the American Society for Testing Materials.<sup>3</sup>

<sup>3</sup> In this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

STANDARD SPECIFICATIONS AND METHODS OF TEST  
FOR  
COTTON GOODS FOR RUBBER AND PYROXYLIN  
COATING<sup>1</sup>



A.S.T.M. Designation: D 334 - 40

ADOPTED, 1934; REVISED, 1940.<sup>2</sup>

Reapproved in 1946 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation D 334; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These specifications and methods of test apply to cotton goods for use in the rubber and pyroxylin coating industry.

#### Quality

2. The goods shall be made of cotton thoroughly cleaned and carded, free from waste, and shall be of good commercial quality, evenly woven.

#### Defects

3. Cotton goods purchased under these specifications will be used in the manufacture of coated fabrics and the seller, in the manufacture and inspection of these goods, shall endeavor to meet the requirements of this industry. All fabrics shall be inspected and burlap on both sides. Certain defects shall be classed as major defects and shall not

occur in a roll on an average of more than one defect to every 15 yd. A minimum allowance of  $\frac{1}{4}$  yd. will be made for each major defect. The following defects are classified as major defects:

Smash	Heavy filling
Washed out oil spots	Shuttle mark
Starchy places	Heavy warp
Group float	Kinky filling
Two contiguous filling threads missing	

NOTE.—These defects are probably not all which should be listed so that this list is subject to change by common agreement. Also, one major defect on the average of every 15 yd. does not represent the quality desired but has been suggested as a fair basis both to manufacturer and to consumer. The aim of the manufacturer will be so to improve their quality that this average yardage between defects may be increased.

The following shall not be present in goods furnished under these specifications:

Oil spots  
Holes  
Torn selvage  
Warp streaks

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1931 to 1934.

**Selvage**

4. Selvages of these fabrics shall be of uniform width and of equal width on both edges of the fabric. Selvages shall be so made that the tension shall be the same as, and shall lie flat with the body of the cloth. Baggy goods and curling selvages shall be cause for rejection.

**Sizing**

5. As little sizing shall be used in the fabric as is necessary for its proper manufacture. Assuming an average content of natural oils and waxes indigenous to the cotton of 4.25 per cent, the total percentage of sizing and natural oils and waxes shall not exceed that given below for each type of fabric:

Sheetings.....	10.75 per cent
Drills.....	12.0 per cent
Sateens.....	12.0 per cent
Broken twills.....	12.0 per cent

**Injurious Chemicals**

6. It shall be recognized that there are certain chemicals, listed below, which are injurious to rubber, and in so far as these come under the control of the manufacturer the quantity shall not exceed the following percentage:

Copper.....	0.001 per cent
Manganese.....	0.0005 per cent
Grease.....	1 per cent

**Width**

7. The average width of the fabric shall be not more than 1.5 per cent over or under the specified width.

**Count**

8. The average count of the fabric shall be not more than 2 per cent in the warp and 5 per cent in the filling over or under the specified count. The average of the readings in a shipment shall not show a variation of over 1 per cent in the warp and 3 per cent in the filling.

**Unit Weight**

9. The unit weight shall be as specified for the fabric with a tolerance from the specified weight of minus 1 per cent on a shipment, minus 2.5 per cent on a roll, and minus 5 per cent on a piece.

**Breaking Strength**

10. (a) The specified breaking strength shall conform to the following requirements. In the case of constructions not *pro rata* to any of those listed below, any specified strength shall be agreed upon when the contract is placed and based on the following breaking strengths:

Construction (Including <i>Pro Ratas</i> )		Breaking Strength, min., lb.	
		Warp	Filling
<b>Sheeting:</b>			
66 in. 48/48	3.00 yd.....	35	25
63 in. 48/48	3.14 yd.....	35	25
60 in. 48/48	3.30 yd.....	35	25
57 in. 48/48	3.47 yd.....	35	25
52 in. 48/48	3.85 yd.....	35	25
50 in. 48/48	4.00 yd.....	35	25
49 in. 48/48	4.05 yd.....	35	25
48 in. 48/48	4.17 yd.....	35	25
<b>Drill:</b>			
59 in.	2.25 yd.....	58	36
66/67 in.	1.87 yd.....	60	40
65 in.	1.93 yd.....	60	40
52 in.	2.38 yd.....	60	40
49 in.	2.53 yd.....	60	40
52 in.	2.20 yd.....	62	48
59 in.	1.85 yd.....	72	43
56 in.	1.93 yd.....	72	43
54 in.	2.00 yd.....	72	43
<b>Twill:</b>			
69 in.	1.37 yd.....	80	59
59 in.	1.60 yd.....	80	59
<b>Broken twill:</b>			
54 in. 85 sley	1.14 yd.....	95	110
56 in.	1.10 yd.....	95	110
54 in. 76/52	1.14 yd.....	100	100
56 in.	1.10 yd.....	100	100
54 in. 85/64	0.98 yd.....	110	130
56 in.	0.95 yd.....	110	130
<b>Sateen:</b>			
54 in. 96 sley	1.55 yd.....	110	50
54 in. 96 sley	1.30 yd.....	110	75
53 in.	1.32 yd.....	110	75
54 in. 96 sley	1.10 yd.....	125	95
53 in.	1.12 yd.....	125	95
54 in. 96 sley	1.05 yd.....	125	120

(b) The average of five determinations in the direction of the warp and five in the direction of the filling shall

be not less than the specified breaking strength for warp and filling respectively.

#### Length of Cuts

11. In respect to length of cuts two classes of delivery may be specified in the purchase contract as follows:

*Class 1.*—Double cuts as far as practicable, and

*Class 2.*—Long cuts.

The proportion of acceptable lengths is given in the following Paragraphs (a) to (c):

(a) *Sheetings:*

*Class 1.*—Ten per cent of pieces in from 40 to 80-yd. lengths will be accepted at the contract price, the balance shall be in pieces not less than 80 yd. in length.

*Class 2.*—The average length of piece shall be 250 yd. with a minimum of 200 yd., the seller having the privilege of including at the contract price up to 10 per cent if made of pieces from 100 to 200 yd. in length.

(b) *Drills, Class 2.*—The average length of piece shall be 175 yd. with a minimum of 120 yd., the seller having the privilege of including at the contract price up to 10 per cent if made of pieces from 60 to 120 yd. in length..

(c) *Sateens, Broken Twills, and Mole-skins, Class 2.*—The minimum length of piece shall be 120 yd. in length, the seller having the privilege of including at the contract price not more than one 60-yd. piece for each 500 yd. shipped.

### METHODS OF TESTING

#### General Methods

12. All test procedures not covered in these specifications shall be in accordance with the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39) of the American Society for Testing Materials.<sup>3</sup>

#### Test Condition

13. The fabrics shall be brought to a standard condition in accordance with Section 2 of Standard Methods D 39<sup>3</sup> prior to testing and tests shall be made under the standard atmospheric conditions.

NOTE.—Tests may be made under prevailing atmospheric conditions except in the settlement of disputes regarding strength and weight.

#### Number of Tests

14. In the acceptance or rejection of each shipment, tests shall be made on samples taken from 20 per cent of the rolls in each shipment; except that in shipments of 1 to 3 rolls, each roll shall be tested; in shipments of 4 to 9 rolls, three rolls shall be tested; in shipments of 10 to 19 rolls, four rolls shall be tested. Five samples shall be taken from each roll selected for purpose of tests on the warp and for tests on the fillings. The seller shall have an opportunity of checking the tests and if agreement cannot be reached, tests shall be made by a referee whose decision shall be final.

#### Sizing

15. (a) Tests to determine the amount of sizing and other nonfibrous materials shall be made in accordance with the following procedure:

(b) *Weighing Bottle.*—Heat a glass weighing bottle of approximately 100-ml. capacity, fitted with a ground-glass cover, in a ventilated drying oven at 105 to 110 C. as follows: Place the bottle and cover separately in the oven and, after drying for 1 hr. or more, remove from the oven and place them in a desiccator and allow to cool to room temperature. Then place the cover on the bottle, and weigh. Repeat the drying, cooling, and weighing until the weight is constant to within plus or

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

minus 0.005 g. This is the "weight of the weighing bottle."

(c) *Test Specimen*.—From the fabric, cut a test specimen weighing approximately 10 g. Particular care shall be taken to prevent the loss of fibers during the treatment. Place the specimen in the weighing bottle and dry in accordance with the procedure described in Paragraph (b). Repeat the drying, cooling, and weighing until the weight is constant to within plus or minus 0.005 g. By subtracting the "weight of the weighing bottle" (Paragraph (b)) from this weight, the weight of the dry specimen, *A*, is obtained.

(d) *Procedure*.—Prepare an approximately 0.1 *N* HCl solution by diluting 8.6 ml. of HCl (sp. gr. 1.19) to 1 liter with distilled water. In a 400-ml. beaker, bring 200 ml. of this solution to boiling and immerse the specimen in the boiling solution. Cover the beaker with a watch glass to minimize loss in volume, and continue the boiling for 30 min. Treat each specimen in a separate beaker. Pour the contents of the beaker onto a fine sieve (No. 1 silk bolting cloth) and wash with running water while squeezing the specimen occasionally with the finger tips. After draining off the water, squeeze the specimen as dry as possible with the fingers, wipe it around the sieve to pick up loose fibers, and immerse it in diluted  $\text{NH}_4\text{OH}$  (10 ml. of  $\text{NH}_4\text{OH}$  (sp. gr. 0.90) diluted to 1 liter with distilled water). Pour the contents of the beaker onto the sieve and wash as directed above. Finally, squeeze the specimen as dry as possible with the fingers, wipe it around the sieve to pick up loose fibers, and place it in the weighing bottle and dry to constant weight in accordance with Paragraph (c). By subtracting the "weight of the weighing bottle" (Paragraph (b)) from this weight, the weight of the dry cotton cellulose, *B*, is obtained.

(e) *Calculation*.—Calculate the total

amount of nonfibrous constituents present as follows:

$$S = \frac{A - B}{A} \times 100$$

where:

*S* = percentage of total sizing, finishing, and other nonfibrous materials (dry basis),

*A* = weight of the dry specimen (Paragraph (c)), and

*B* = weight of the dry cotton cellulose (Paragraph (d)).

NOTE.—The result obtained by this procedure includes not only the starch, filling, and finishing materials added during manufacturing, but also some of the waxes and other non-cellulose natural constituents of raw cotton. The natural impurities and the amounts removed by this treatment vary with the grade, source, and previous treatment of the cotton. When it is desired to determine the actual amount of sizing or other added substance, the actual loss due to removal of natural constituents of the cotton should be determined on the unsized cotton yarn used in making the fabric. If the unsized yarn is not available, then an allowance of 4.25 per cent may be made as the amount of natural noncellulose materials removed.

### Copper and Manganese

16. The copper and manganese contents shall be determined in accordance with the Standard Method of Test for Colorimetric Determination of Small Amounts of Copper and Manganese in Textiles (A.S.T.M. Designation: D 377) of the American Society for Testing Materials.<sup>3</sup>

### Width

17. The width of the fabric in any roll or piece shall be determined by laying the fabric on a flat surface, under no tension, with wrinkles removed, and measuring the width perpendicular to the selvage, at five different places evenly distributed through the roll or piece. The average of these five measurements shall be considered the actual width.

**Count**

18. The count of the fabric in any roll or piece shall be determined by counting a space of not less than 1 in. in at least five different places in the roll or piece without duplicating the determination on any one set of threads. The average of these five or more determinations in the warp and filling shall be the count. No determination shall be taken nearer the selvage than one-tenth the width of the fabric.

**Unit Weight**

19. The unit weight shall be expressed in linear yards of the fabric per pound and shall be determined from the invoiced yardage and the weight of the entire roll or piece (both verified when necessary).

**Breaking Strength**

20. The breaking strength shall be determined by the grab method as described in Section 10 of Standard Methods D 39,<sup>3</sup> and in accordance with the following requirements:

The back jaw of each clamp shall be 2 in. or more in width and the front jaw shall be 1 in. in width. The jaws shall be smooth and flat with edges slightly rounded to prevent cutting. The initial distance between clamps of the testing machine shall be 3 in. and the pulling clamp shall travel at a uniform rate of  $12 \pm \frac{1}{2}$  in. per min. Test specimens shall be 6 in. in length by 4 in. in width and no two specimens shall include the same yarns.

# *Standard Specifications for*

## INSULATED WIRE AND CABLE: HEAT-RESISTING RUBBER COMPOUND<sup>1</sup>



**A.S.T.M. Designation: D 469 - 46**

ADOPTED, 1941; REVISED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 469; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These specifications cover wire and cable insulated with a vulcanized rubber compound. The suitability of the compound as insulation shall be determined solely by physical and electrical tests. The compound shall be called heat-resisting rubber compound. This compound is ordinarily recommended for use at conductor temperatures not in excess of 75 C.

(b) Except for the rubber insulation, wire and cable supplied under these specifications shall conform to the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (A.S.T.M. Designation: D 27) of the American Society for Testing Materials.<sup>3</sup>

### Methods of Test

2. All measurements and tests necessary for determining the conformity of the insulated wire and cable with these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications, including the use of a temperature of 80 C. in the oxygen pressure chamber aging test.

ance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications, including the use of a temperature of 80 C. in the oxygen pressure chamber aging test.

### Physical Properties

3. The insulation shall consist of a properly vulcanized rubber compound which shall conform to the requirements as to physical properties prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min., psi. ....	1500
Tensile stress at 200 per cent elongation, min., psi. ....	400
Elongation at rupture, min., per cent. ....	400
Set in 2-in. gage length, max., in. ....	$\frac{1}{8}$
Tensile strength after 20 hr. air pressure heat test, min., psi. ....	1000
Tensile strength after 20 hr. air pressure heat test, min., per cent of the original value. ....	50
Elongation at rupture after 20 hr. air pressure heat test, min., per cent. ....	300
Elongation at rupture after 20 hr. air pressure heat test, min., per cent of the original value. ....	65
Tensile strength and elongation at rupture after 168 hr., at 80 C. and 300-lb. pressure, oxygen pressure test, min., per cent of the original value. ....	75

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1937 to 1941, being revised in 1939, 1940, and 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For nonleaded submarine cables,  $\frac{1}{2}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits except where one side of a delta system is permanently grounded, in which case, multiply the circuit voltage (phase to phase) by 1.73 and use the resulting voltage to select the corresponding insulation thickness.

NOTE 5.—Above 5000 v. alternating-current, ozone-resistant insulation is recommended as prescribed in the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (A.S.T.M. Designation: D 574) of the American Society for Testing Materials.<sup>2</sup>NOTE 6.—Where the National Electrical Code has jurisdiction, sizes Nos. 14, 12, and 10 A. w. g. with  $\frac{3}{8}$ -in. thickness of insulation and size No. 8 A. w. g. with  $\frac{3}{4}$ -in. thickness of insulation rated at 600 v. may be used.

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thick- ness on Each Conductor, sixty- fourths of an inch	Test Voltage
0 to 300 .....	No. 18 to No. 16 .....	2	1 000
	No. 16 to No. 9 <sup>a</sup> .....	3	3 000
	No. 8 to No. 2 .....	4	3 500
	No. 1 to No. 0000 .....	5	4 000
0 to 600 .....	225 000 to 500 000 cir. mils. ....	6	5 000
	525 000 to 1 000 000 cir. mils. ....	7	6 000
	Over 1 000 000 cir. mils. ....	8	7 000
601 to 1000 .....	No. 14 to No. 8 .....	4	5 000
	No. 7 to No. 2 .....	5	6 000
	No. 1 to No. 0000 .....	6	7 500
	225 000 to 500 000 cir. mils. ....	7	9 000
1001 to 2000 .....	525 000 to 1 000 000 cir. mils. ....	8	10 000
	Over 1 000 000 cir. mils. ....	9	11 000
	No. 14 to No. 8 .....	5	6 000
2001 to 3000 .....	No. 7 to No. 2 .....	6	7 500
	No. 1 to No. 0000 .....	7	9 000
	225 000 to 500 000 cir. mils. ....	8	10 000
	525 000 to 1 000 000 cir. mils. ....	9	11 000
3001 to 4000 .....	Over 1 000 000 cir. mils. ....	9	11 000
	No. 10 to No. 8 .....	7	9 000
	No. 7 to No. 2 .....	8	10 000
4001 to 5000 .....	No. 1 to No. 0000 .....	8	10 000
	225 000 to 500 000 cir. mils. ....	9	11 000
	525 000 to 1 000 000 cir. mils. ....	9	11 000
	Over 1 000 000 cir. mils. ....	10	12 500
5001 to 6000 .....			
	No. 10 to No. 8 .....	9	11 000
	No. 7 to No. 2 .....	9	11 000
	No. 1 to No. 0000 .....	9	11 000
6001 to 7000 .....	225 000 to 500 000 cir. mils. ....	10	12 500
	525 000 to 1 000 000 cir. mils. ....	10	12 500
	Over 1 000 000 cir. mils. ....	11	13 500
7001 to 8000 .....	No. 8 .....	10	12 500
	No. 7 to No. 2 .....	10	12 500
	No. 1 to No. 0000 .....	10	12 500
	225 000 to 500 000 cir. mils. ....	11	13 500
8001 to 9000 .....	525 000 to 1 000 000 cir. mils. ....	11	13 500
	Over 1 000 000 cir. mils. ....	12	15 000
	No. 8 to No. 0000 .....	10	12 500
9001 to 10 000 .....	225 000 to 1 000 000 cir. mils. ....	11	13 500
	Over 1 000 000 cir. mils. ....	12	15 000
	No. 8 to No. 0000 .....	12	15 000
10 001 to 11 000 .....	225 000 to 1 000 000 cir. mils. ....	12	15 000
	Over 1 000 000 cir. mils. ....	13	16 500
	No. 8 to No. 0000 .....	11	13 500
11 001 to 12 000 .....	225 000 to 1 000 000 cir. mils. ....	11	13 500
	Over 1 000 000 cir. mils. ....	12	15 000
	No. 8 to No. 0000 .....	14	17 500
12 001 to 13 000 .....	225 000 to 1 000 000 cir. mils. ....	14	17 500
	Over 1 000 000 cir. mils. ....	15	19 000
	No. 8 to No. 0000 .....	12	15 000
13 001 to 14 000 .....	225 000 to 1 000 000 cir. mils. ....	12	15 000
	Over 1 000 000 cir. mils. ....	13	16 500
	No. 8 to No. 0000 .....	16	20 000
14 001 to 15 000 .....	225 000 to 1 000 000 cir. mils. ....	16	20 000
	Over 1 000 000 cir. mils. ....	17	21 000
	No. 8 to No. 0000 .....	16	20 000

<sup>a</sup> Sizes Nos. 16 and 18 are not recognized by the National Electrical Code for 600-v. service.



## High Voltage Test

4. Each coil, reel, or length of wire or cable after vulcanization shall successfully withstand the application of an alternating-current voltage of not less than that prescribed in Table II for a period of 5 min.

## Insulation Resistance

5. (a) Each coil, reel, or length of wire or cable after being subjected to the high voltage test specified in Section 4 shall have an insulation resistance not less than that prescribed in Table III.

(b) In calculating insulation resist-

TABLE III.—INSULATION RESISTANCE.

Size of Conductor, A.w.g. numbers or circular mils	Solid or Stranded	Dia- meter, in.	Insulation Thickness in Sixty-fourths of an Inch											
			2	3	4	5	6	7	8	9	10	11	12	
			Insulation Resistance, megohms—1000 ft. at 60 F. (15.5 C.)											
No. 18.....	Solid	0.040	4 300	5 550	....	....	....	....	....	....	....	....	....	
No. 16.....	Solid	0.051	3 650	4 750	....	....	....	....	....	....	....	....	....	
No. 14.....	Solid	0.064	....	4 150	4 950	5 650	6 280	6 880	7 300	7 730	8 130	8 490	8 830	
No. 12.....	Solid	0.081	....	3 530	4 280	4 930	5 500	6 000	6 450	6 880	7 250	7 600	7 986	
No. 10.....	Solid	0.102	....	3 000	3 680	4 250	4 800	5 250	5 680	6 050	6 430	6 770	7 080	
No. 9.....	Solid	0.114	....	2 800	3 390	3 950	4 450	4 900	5 310	5 690	6 040	6 360	6 670	
No. 8.....	Solid	0.128	....	....	3 100	3 650	4 150	4 580	4 980	5 380	5 680	5 980	6 280	
No. 7.....	Solid	0.144	....	....	2 860	3 370	3 818	4 230	4 610	4 960	5 290	5 590	5 870	
No. 6.....	Stranded	0.162	....	....	2 380	2 800	3 200	3 600	3 950	4 250	4 550	4 780	5 100	
No. 4.....	Stranded	0.204	....	....	1 950	2 350	2 730	3 050	3 350	3 630	3 900	4 170	4 430	
No. 2.....	Stranded	0.258	....	....	1 630	1 950	2 280	2 680	2 830	3 080	3 330	3 570	3 800	
No. 1.....	Stranded	0.289	....	....	....	1 780	2 050	2 330	2 580	2 800	3 050	3 260	3 480	
No. 0.....	Stranded	0.325	....	....	....	1 600	1 880	2 100	2 350	2 580	2 780	3 000	3 180	
No. 00.....	Stranded	0.365	....	....	....	1 450	1 700	1 930	2 150	2 350	2 550	2 760	2 930	
No. 000.....	Stranded	0.410	....	....	....	1 300	1 550	1 750	1 950	2 130	2 330	2 510	2 700	
No. 0000.....	Stranded	0.460	....	....	....	1 200	1 400	1 580	1 780	1 950	2 100	2 300	2 450	
250 000 cir. mils.....	Stranded	0.575	....	....	....	....	1 300	1 480	1 650	1 830	2 000	2 150	2 300	
350 000 cir. mils.....	Stranded	0.681	....	....	....	....	1 130	1 280	1 430	1 580	1 730	1 870	2 000	
500 000 cir. mils.....	Stranded	0.814	....	....	....	....	....	950	1 100	1 230	1 350	1 480	1 620	
750 000 cir. mils.....	Stranded	0.998	....	....	....	....	....	....	900	1 030	1 130	1 250	1 360	
1 000 000 cir. mils.....	Stranded	1.152	....	....	....	....	....	....	800	900	1 000	1 100	1 190	
1 250 000 cir. mils.....	Stranded	1.289	....	....	....	....	....	....	800	900	1 000	1 090	1 180	
1 500 000 cir. mils.....	Stranded	1.412	....	....	....	....	....	....	750	825	925	1 000	1 080	
1 750 000 cir. mils.....	Stranded	1.526	....	....	....	....	....	....	700	775	850	950	1 000	
2 000 000 cir. mils.....	Stranded	1.631	....	....	....	....	....	....	650	750	800	880	950	

Size of Conductor, A.w.g. numbers or circular mils	Solid or Stranded	Diameter, in.	Insulation Thickness in Sixty-fourths of an Inch				
			13	14	15	16	17
			Insulation Resistance, megohms—1000 ft. at 60 F. (15.5 C.)				
No. 8.....	Solid	0.128	6 550	6 800	7 050	7 300	7 450
No. 7.....	Solid	0.144	6 140	6 890	6 630	6 860	7 080
No. 6.....	Stranded	0.162	5 330	5 580	5 750	6 000	6 230
No. 4.....	Stranded	0.204	4 650	4 850	5 050	5 250	5 450
No. 2.....	Stranded	0.258	3 480	4 200	4 400	4 580	4 750
No. 1.....	Stranded	0.289	3 680	3 830	4 000	4 200	4 380
No. 0.....	Stranded	0.325	3 380	3 530	3 700	3 900	4 050
No. 00.....	Stranded	0.365	3 100	3 250	3 450	3 580	3 750
No. 000.....	Stranded	0.410	2 850	3 080	3 180	3 330	3 450
No. 0000.....	Stranded	0.460	2 600	2 750	2 900	3 030	3 180
250 000 cir. mils.....	Stranded	0.575	2 450	2 600	2 730	2 880	2 980
350 000 cir. mils.....	Stranded	0.681	2 130	2 250	2 350	2 480	2 600
500 000 cir. mils.....	Stranded	0.814	1 850	1 980	2 080	2 180	2 280
750 000 cir. mils.....	Stranded	0.998	1 550	1 650	1 750	1 850	1 950
1 000 000 cir. mils.....	Stranded	1.152	1 380	1 480	1 550	1 650	1 730
1 250 000 cir. mils.....	Stranded	1.289	1 250	1 330	1 430	1 500	1 580
1 500 000 cir. mils.....	Stranded	1.412	1 150	1 230	1 300	1 350	1 480
1 750 000 cir. mils.....	Stranded	1.526	1 080	1 150	1 230	1 300	1 380
2 000 000 cir. mils.....	Stranded	1.631	1 030	1 100	1 150	1 230	1 300

NOTE 1.—This table is based on a constant,  $K = 10,560$  in the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where:

$R$  = insulation resistance in megohms—1000 ft.,

$K$  = constant,

$D$  = diameter over insulation, on each conductor, and

$d$  = diameter over conductor.

NOTE 2.—For an intermediate size, the insulation resistance shall be that of the next larger size.

ance the temperature coefficients given in Table IV shall be used.

#### Moisture Absorption

6. (a) The increase in the specific inductive capacity of the insulation shall be used as an indication of the amount of moisture absorbed.

(b) The specific inductive capacity after 24-hr. immersion in water shall not exceed five. The increase between the ends of the first and the fourteenth days shall not exceed 20 per cent and that between the ends of the seventh and

for insulations less than  $\frac{1}{8}$  in. in thickness and not less than 90 per cent of the specified thickness for insulations of  $\frac{1}{8}$  in. and greater.

(b) The thicknesses prescribed in Table II apply to single-conductor cable and the individual conductors of multiple-conductor cables, leaded or braided, except special applications such as aerial, non-leaded submarine, and vertical riser cable.

NOTE.—In the Appendix will be found an interpretation of the insulation thicknesses given in Table II.

TABLE IV.—TEMPERATURE COEFFICIENTS.

Temperature			Temperature		
Fahren- heit Deg.	Centi- grade Deg.	Tem- pera- ture Coeffi- cient	Fahren- heit Deg.	Centi- grade Deg.	Tem- pera- ture Coeffi- cient
46	7.8...	0.65	61	16.1...	1.03
47	8.3...	0.67	62	16.7...	1.07
48	8.9...	0.69	63	17.2...	1.10
49	9.4...	0.71	64	17.8...	1.13
50	10.0...	0.73	65	18.3...	1.17
51	10.6...	0.76	66	18.9...	1.20
52	11.1...	0.78	67	19.4...	1.24
53	11.7...	0.80	68	20.0...	1.28
54	12.2...	0.83	69	20.6...	1.32
55	12.8...	0.86	70	21.1...	1.36
56	13.3...	0.88	71	21.7...	1.40
57	13.9...	0.91	72	22.2...	1.45
58	14.4...	0.94	73	22.8...	1.50
59	15.0...	0.97	74	23.3...	1.55
60	15.6...	1.00	75	23.9...	1.59

fourteenth days shall not exceed 5 per cent.

(c) The specific inductive capacity shall be determined at either 1000 or 60 cycles. Where measured at 1000 cycles, the voltage impressed upon the conductor shall not exceed 10 v. Where measured at 60 cycles, the voltage impressed upon the conductor shall be equivalent to a stress of between 30 to 40 v. per mil of insulation.

#### Thickness

7. (a) The average thickness of the insulation shall be not less than that prescribed in Table II. The minimum thickness shall be not less than 95 per cent of the thickness required by Table II

(c) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

(d) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement is made by "slicing off" the heavier side of the insulation.) In the case of multiple-conductor cable, the

measurements shall be made on the individual wires before being cabled.

### Workmanship

8. (a) The insulation shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(b) *Shielding*.—Wires and cables not supported on insulators shall be shielded or suitably protected against corona in accordance with the detailed recommendations contained in Sections 20 to 24, inclusive, of Tentative Specifications D 27.<sup>3</sup>

(c) *Covering*.—The insulation of braided wires up to No. 7 A.w.g., inclusive, shall be covered with a single cotton braid. On larger sizes the fibrous covering shall consist of a cable tape and cotton braid or two cotton braids. The cable tape may be applied before vulcanization. In multiple-conductor cables the individual conductors shall not be braid-covered unless specified.

(d) *Repairs and Joints*.—When repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and

mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 7.

### Rejection

9. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

(b) When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

(c) Each coil, reel, or length which fails to conform to the electrical requirements of these specifications shall be rejected.

(See Appendix, p. 351)

## APPENDIX

## INTERPRETATION OF INSULATION THICKNESSES IN TABLE II

For series-street-lighting cable, use the following procedure:

A1. (a) If the transformer is not equipped with protectors, use the open-circuit secondary voltage of the transformer as rated voltage of the cable and select the proper wall thickness from Table II.

(b) If the transformer is equipped with protectors use the closed or loaded secondary voltage of the transformer as the rated-circuit voltage of the cable and select the proper wall thickness from Table II.

NOTE.—It is preferable, both in Paragraphs (a) and (b) above, to select a cable with a higher voltage rating than obtained using the above procedure, both as a measure of safety and to allow the substitution of a larger transformer without necessitating the replacement of the cable.

The thickness of insulation for the various systems shall be determined as follows:

A2. (a) For three-phase systems with grounded or ungrounded neutral, use thickness values in accordance with Table II.

(b) For single or two-phase systems up to and including 5000 v., use thickness values in accordance with Table II, as specified for grounded neutral. Where it is not definitely

specified that a line operates as an isolated single or two-phase system, it shall be considered as a branch of a grounded three-phase circuit and the thickness of insulation shall be that required by the phase-to-phase voltage of this grounded, three-phase circuit as given in Table II.

(c) For single or two-phase systems operating at over 5000 v. with one side grounded, multiply the circuit voltage (phase-to-phase) by 1.73 and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table II.

(d) For single or two-phase systems operating at over 5000 v. with the center grounded, multiply the circuit voltage (phase-to-phase) by 0.866 ( $\frac{1}{2} \sqrt{3}$ ) and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table II.

(e) For single or two-phase ungrounded systems operating at over 5000 v., multiply the circuit voltage (phase-to-phase) by 0.866 ( $\frac{1}{2} \sqrt{3}$ ) and use the resulting voltage value to select the corresponding insulation thickness for ungrounded neutral as specified in Table II.

(f) For direct-current systems, consider the same as single-phase alternating-current systems in accordance with Paragraph A2 (b) above.

# *Standard Specifications for*

## INSULATED WIRE AND CABLE: PERFORMANCE RUBBER COMPOUND<sup>1</sup>



A.S.T.M. Designation: D 353 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 353; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These specifications cover wire and cable insulated with a vulcanized rubber compound. The suitability of the compound as insulation shall be determined solely by physical and electrical tests. The compound shall be called performance rubber compound.

(b) Except for the rubber insulation, wire and cable supplied under these specifications shall conform to the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (A.S.T.M. Designation: D 27) of the American Society for Testing Materials.<sup>3</sup>

### Methods of Test

2. All measurements and tests necessary for determining the conformity of the insulated wire and cable with these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable

(A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications.

### Physical Properties

3. The insulation shall consist of a properly vulcanized rubber compound which shall conform to the requirements as to physical properties prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min., psi. ....	1200
Tensile stress at 200 per cent elongation min., psi. ....	300
Elongation at rupture, min., per cent. ....	400
Set in 2-in. gage length, max., in. ....	$\frac{1}{8}$
Depreciation in tensile strength and elongation after 96 hr. in air oven test at 157 to 159 F., (69.4 to 70.6 C.), max., per cent. ....	15
Depreciation in tensile strength and elongation after 96 hr. in oxygen pressure test, max., per cent. ....	25

### High Voltage Test

4. Each coil, reel, or length of wire or cable after vulcanization shall successfully withstand the application of an alternating-current voltage of not less than that prescribed in Table II for a period of 5 min.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these specifications were published as tentative from 1932 to 1941, being revised in 1933, 1934, 1935, 1936, 1937, 1939, 1940, and 1941.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For nonleaded submarine cables,  $\frac{1}{8}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits except where one side of a delta system is permanently grounded, in which case, multiply the circuit voltage (phase to phase) by 1.73 and use the resulting voltage to select the corresponding insulation thickness.

NOTE 5.—Above 5000 v. alternating-current, ozone-resistant insulation is recommended as prescribed in the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (A.S.T.M. Designation: D 574) of the American Society for Testing Materials.\*

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thick- ness on Each Conductor, sixty- fourths of an inch	Test Voltage
0 to 300.....	No. 18 to No. 16.....	2	1 000
	No. 16 to No. 9 <sup>a</sup> .....	3	3 000
	No. 8 to No. 2.....	4	3 500
	No. 1 to No. 0000.....	5	4 000
0 to 600.....	225 000 to 500 000 cir. mils.....	6	5 000
	525 000 to 1 000 000 cir. mils.....	7	6 000
	Over 1 000 000 cir. mils.....	8	7 000
	No. 14 to No. 8.....	4	5 000
	No. 7 to No. 2.....	5	6 000
	No. 1 to No. 0000.....	6	7 500
601 to 1000.....	225 000 to 500 000 cir. mils.....	7	9 000
	525 000 to 1 000 000 cir. mils.....	8	10 000
	Over 1 000 000 cir. mils.....	9	11 000
	No. 14 to No. 8.....	5	6 000
	No. 7 to No. 2.....	6	7 500
	No. 1 to No. 0000.....	7	9 000
1001 to 2000.....	225 000 to 500 000 cir. mils.....	8	10 000
	525 000 to 1 000 000 cir. mils.....	9	11 000
	Over 1 000 000 cir. mils.....	9	11 000
	No. 10 to 8.....	7	9 000
	No. 7 to No. 2.....	8	10 000
	No. 1 to No. 0000.....	8	10 000
2001 to 3000.....	225 000 to 500 000 cir. mils.....	9	11 000
	525 000 to 1 000 000 cir. mils.....	9	11 000
	Over 1 000 000 cir. mils.....	10	12 500
	No. 10 to No. 8.....	9	11 000
	No. 7 to No. 2.....	9	11 000
	No. 1 to No. 0000.....	9	11 000
3001 to 4000.....	225 000 to 500 000 cir. mils.....	10	12 500
	525 000 to 1 000 000 cir. mils.....	10	12 500
	Over 1 000 000 cir. mils.....	11	13 500
	No. 8.....	10	12 500
	No. 7 to No. 2.....	10	12 500
	No. 1 to No. 0000.....	10	12 500
4001 to 5000.....	225 000 to 500 000 cir. mils.....	11	13 500
	525 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	10	12 500
5001 to 6000.....	225 000 to 1 000 000 cir. mils.....	11	13 500
(grounded).....	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	12	15 000
5001 to 6000.....	225 000 to 1 000 000 cir. mils.....	12	15 000
(ungrounded).....	Over 1 000 000 cir. mils.....	13	16 500
	No. 8 to No. 0000.....	11	13 500
6001 to 7000.....	225 000 to 1 000 000 cir. mils.....	11	13 500
(grounded).....	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	14	17 500
6001 to 7000.....	225 000 to 1 000 000 cir. mils.....	14	17 500
(ungrounded).....	Over 1 000 000 cir. mils.....	15	19 000
	No. 8 to No. 0000.....	12	15 000
7001 to 8000.....	225 000 to 1 000 000 cir. mils.....	12	15 000
(grounded).....	Over 1 000 000 cir. mils.....	13	16 500
	No. 8 to No. 0000.....	16	20 000
7001 to 8000.....	225 000 to 1 000 000 cir. mils.....	16	20 000
(ungrounded).....	Over 1 000 000 cir. mils.....	17	21 000

\* Sizes Nos. 16 and 18 are not recognized by the National Electrical Code for 600-v. service.

TABLE III.—INSULATION RESISTANCE.

Insulation Thickness in Sixty-fourths of an Inch.....			2	3	4	5	6	7	8	9	10	12
Size of Conductor, A. w. g. numbers or circular mils	Solid or Stranded	Diam- eter, in.	Insulation Resistance, megohms—1000 ft. at 60 F. (15.5 C.)									
No. 18.....	Solid	0.040	8600	11 100	.....	.....	.....	.....	.....	.....	.....	.....
No. 16.....	Solid	0.051	7300	9 500	.....	.....	.....	.....	.....	.....	.....	.....
No. 14.....	Solid	0.064	.....	8 300	9900	11 300	12 550	13 650	14 600	15 450	16 250	17 650
No. 12.....	Solid	0.081	.....	7 050	8550	9 850	11 000	12 000	12 900	13 750	14 500	15 850
No. 10.....	Solid	0.102	.....	6 000	7350	8 500	9 600	10 500	11 350	12 100	12 850	14 150
No. 8.....	Solid	0.128	.....	.....	6200	7 300	8 300	9 150	9 950	10 650	11 350	12 550
No. 6.....	Stranded	0.184	.....	4750	5 600	6 400	7 200	7 900	8 500	9 100	10 200	.....
No. 4.....	Stranded	0.232	.....	3900	4 700	5 450	6 100	6 700	7 250	7 800	8 850	.....
No. 2.....	Stranded	0.292	.....	.....	3250	3 900	4 550	5 150	5 650	6 150	6 650	7 600
No. 1.....	Stranded	0.332	.....	.....	.....	3 550	4 100	4 650	5 150	5 600	6 100	6 950
No. 0.....	Stranded	0.373	.....	.....	.....	3 200	3 750	4 200	4 700	5 150	5 550	6 350
No. 00.....	Stranded	0.418	.....	.....	.....	2 900	3 400	3 850	4 300	4 700	5 100	5 850
No. 000.....	Stranded	0.470	.....	.....	.....	2 600	3 100	3 500	3 900	4 250	4 650	5 400
No. 0000.....	Stranded	0.528	.....	.....	.....	2 400	2 800	3 150	3 550	3 900	4 200	4 900
250 000 cir. mils.....	Stranded	0.575	.....	.....	.....	.....	2 600	2 950	3 300	3 650	4 000	4 600
350 000 cir. mils.....	Stranded	0.681	.....	.....	.....	.....	2 250	2 550	2 850	3 150	3 450	4 000
500 000 cir. mils.....	Stranded	0.814	.....	.....	.....	.....	1 900	2 200	2 450	2 700	2 950	3 450
750 000 cir. mils.....	Stranded	0.998	.....	.....	.....	.....	.....	1 800	2 050	2 250	2 500	2 900
1 000 000 cir. mils.....	Stranded	1.152	.....	.....	.....	.....	.....	1 600	1 800	2 000	2 200	2 600
1 250 000 cir. mils.....	Stranded	1.289	.....	.....	.....	.....	.....	.....	1 600	1 800	2 000	2 350
1 500 000 cir. mils.....	Stranded	1.412	.....	.....	.....	.....	.....	.....	1 500	1 650	1 850	2 150
1 750 000 cir. mils.....	Stranded	1.526	.....	.....	.....	.....	.....	.....	1 400	1 550	1 700	2 000
2 000 000 cir. mils.....	Stranded	1.631	.....	.....	.....	.....	.....	.....	1 300	1 500	1 600	1 900

Insulation Thickness in Sixty-fourths of an Inch.....			13	14	15	16	17
Size of Conductor, A. w. g. numbers or circular mils	Solid or Stranded	Diameter, in.	Insulation Resistance, megohms—1000 ft. at 60 F. (15.5 C.)				
No. 8.....	Solid	0.128	13 100	13 600	14 100	14 600	14 900
No. 6.....	Stranded	0.184	10 650	11 150	11 500	12 000	12 450
No. 4.....	Stranded	0.232	9 300	9 700	10 100	10 500	10 900
No. 2.....	Stranded	0.292	7 950	8 400	8 800	9 150	9 500
No. 1.....	Stranded	0.332	7 350	7 650	8 000	8 400	8 750
No. 0.....	Stranded	0.373	6 750	7 050	7 400	7 800	8 100
No. 00.....	Stranded	0.418	6 200	6 500	6 900	7 150	7 500
No. 000.....	Stranded	0.470	5 700	6 150	6 350	6 650	6 900
No. 0000.....	Stranded	0.528	5 200	5 500	5 800	6 050	6 350
250 000 cir. mils.....	Stranded	0.575	4 900	5 200	5 450	5 750	5 950
350 000 cir. mils.....	Stranded	0.681	4 250	4 500	4 700	4 950	5 200
500 000 cir. mils.....	Stranded	0.814	3 700	3 950	4 150	4 350	4 550
750 000 cir. mils.....	Stranded	0.998	3 100	3 300	3 500	3 700	3 900
1 000 000 cir. mils.....	Stranded	1.152	2 750	2 950	3 100	3 300	3 450
1 250 000 cir. mils.....	Stranded	1.289	2 500	2 650	2 850	3 000	3 150
1 500 000 cir. mils.....	Stranded	1.412	2 300	2 450	2 600	2 700	2 950
1 750 000 cir. mils.....	Stranded	1.526	2 150	2 300	2 450	2 600	2 750
2 000 000 cir. mils.....	Stranded	1.631	2 050	2 200	2 300	2 450	2 600

NOTE 1.—This table is based on a constant,  $K = 21,120$  in the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where  $R$  = the insulation resistance in megohms—1000 ft., $K$  = constant, $D$  = diameter over insulation, on each conductor, and $d$  = diameter over conductor.

NOTE 2.—For an intermediate size, the insulation resistance shall be that of the next larger size.

### Insulation Resistance

5. (a) Each coil, reel, or length of wire or cable after being subjected to the high voltage test specified in Section 4 shall have an insulation resistance not less than that prescribed in Table III.

(b) In calculating insulation resistance the temperature coefficients given in Table IV shall be used.

TABLE IV.—TEMPERATURE COEFFICIENTS.

Temperature		Temperature Coefficient	Temperature		Temperature Coefficient
Fahrenheit Deg.	Centigrade Deg.		Fahrenheit Deg.	Centigrade Deg.	
46	7.8...	0.69	61	16.1...	1.03
47	8.3...	0.71	62	16.7...	1.05
48	8.9...	0.73	63	17.2...	1.08
49	9.4...	0.75	64	17.8...	1.11
50	10.0...	0.77	65	18.3...	1.14
51	10.6...	0.79	66	18.9...	1.17
52	11.1...	0.81	67	19.4...	1.20
53	11.7...	0.83	68	20.0...	1.23
54	12.2...	0.85	69	20.6...	1.26
55	12.8...	0.88	70	21.1...	1.30
56	13.3...	0.90	71	21.7...	1.33
57	13.9...	0.92	72	22.2...	1.37
58	14.4...	0.94	73	22.8...	1.40
59	15.0...	0.97	74	23.3...	1.44
60	15.6...	1.00	75	23.9...	1.48

### Moisture Absorption

6. (a) The increase in the specific inductive capacity of the insulation shall be used as an indication of the amount of moisture absorbed.

(b) The specific inductive capacity after 24-hr. immersion in water shall not exceed six. The increase between the ends of the first and the fourteenth days shall not exceed 20 per cent and that between the ends of the seventh and fourteenth days shall not exceed 5 per cent.

(c) The specific inductive capacity shall be determined at either 1000 or 60 cycles. Where measured at 1000 cycles, the voltage impressed upon the conductor shall not exceed 10 v. Where measured at 60 cycles, the voltage impressed upon the conductor shall be equivalent to a stress of between 30 to 40 v. per mil of insulation.

### Thickness

7. (a) The average thickness of the insulation shall be not less than that prescribed in Table II. The minimum thickness shall be not less than 95 per cent of the thickness required by Table II for insulations less than  $\frac{1}{8}$  in. in thickness and not less than 90 per cent of the specified thickness for insulations of  $\frac{1}{8}$  in. and greater.

(b) The thicknesses prescribed in Table II apply to single-conductor cable and the individual conductors of multiple-conductor cables, leaded or braided, except special applications such as aerial, non-leaded submarine, and vertical riser cable.

NOTE.—In the Appendix will be found an interpretation of the insulation thicknesses given in Table II.

(c) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

(d) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference



between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement is made by "slicing off" the heavier side of the insulation.) In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

### Workmanship

8. (a) The insulation shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(b) *Shielding*.—Wires and cables not supported on insulators shall be shielded or suitably protected against corona in accordance with the detailed recommendations contained in Sections 20 to 24, inclusive, of A.S.T.M. Specifications D 27.<sup>3</sup>

(c) *Covering*.—The insulation of braided wires up to No. 7 A.w.g., inclusive, shall be covered with a single cotton braid. On larger sizes the fibrous covering shall consist of a cable tape and cotton braid or two cotton braids. The cable tape may be applied before vulcanization. In multiple-conductor cables the individual conductors shall not be braid-covered unless specified.

(d) *Repairs and Joints*.—When repairs or joints are made in the insulation,

the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 7.

### Rejection

9. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

(b) When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

(c) Each coil, reel, or length which fails to conform to the electrical requirements of these specifications shall be rejected.

(See Appendix, p. 357)

## APPENDIX

## INTERPRETATION OF INSULATION THICKNESSES IN TABLE II

For series-street-lighting cable, use the following procedure:

A1. (a) If the transformer is not equipped with protectors, use the open-circuit secondary voltage of the transformer as rated voltage of the cable and select the proper wall thickness from Table II.

(b) If the transformer is equipped with protectors use the closed or loaded secondary voltage of the transformer as the rated-circuit voltage of the cable and select the proper wall thickness from Table II.

NOTE.—It is preferable, both in Paragraphs (a) and (b) above, to select a cable with a higher voltage rating than obtained using the above procedure, both as a measure of safety and to allow the substitution of a larger transformer without necessitating the replacement of the cable.

The thickness of insulation for the various systems shall be determined as follows:

A2. (a) For three-phase systems with grounded or ungrounded neutral, use thickness values in accordance with Table II.

(b) For single or two-phase systems up to and including 5000 v., use thickness values in accordance with Table II, as specified for grounded neutral. Where it is not definitely specified that a line operates as an isolated

single or two-phase system, it shall be considered as a branch of a grounded three-phase circuit and the thickness of insulation shall be that required by the phase-to-phase voltage of this grounded, three-phase circuit as given in Table II.

(c) For single or two-phase systems operating at over 5000 v. with one side grounded, multiply the circuit voltage (phase-to-phase) by 1.73 and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table II.

(d) For single or two-phase systems operating at over 5000 v. with the center grounded, multiply the circuit voltage (phase-to-phase) by  $0.866 (\frac{1}{2} \sqrt{3})$  and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table II.

(e) For single or two-phase ungrounded systems operating at over 5000 v., multiply the circuit voltage (phase-to-phase) by  $0.866 (\frac{1}{2} \sqrt{3})$  and use the resulting voltage value to select the corresponding insulation thickness for ungrounded neutral as specified in Table II.

(f) For direct-current systems, consider the same as single-phase alternating-current systems in accordance with Paragraph A2 (b) above.

# *Standard Methods of Test for*

## VISCOSITY AND TOTAL SOLIDS CONTENT OF RUBBER CEMENTS<sup>1</sup>



A.S.T.M. Designation: D 553 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 553; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods of test cover procedures for determining the viscosity and total solids content of rubber cements containing organic solvents as differentiated from latex cements or dispersions of rubber in water.

### TOTAL SOLIDS

#### Procedure

2. Approximately 10 g. of the sample shall be poured into a low-form weighing bottle, covered, and weighed. After removing the cover, heat shall be applied at 70 C. until the sample reaches constant weight. The sample shall then be cooled in a desiccator and weighed.

#### Calculations

3. The percentage of total solids shall be calculated as follows:

$$\text{Total solids, per cent} = \frac{\text{wt. of residue}}{\text{wt. of sample}} \times 100$$

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1939 to 1942.

### VISCOSITY

#### Viscosity Tests

4. The following alternate procedures for determining viscosity are included:

*Funnel Method.*—This method gives closely duplicable results but is limited in use to those cements having viscosities which permit sufficiently rapid flow through the funnel to avoid excessive loss of solvent.

*Falling Cylinder Method.*—This method is adapted to rubber cements having a wider range of viscosities and offers some advantages in control testing due to ease of manipulation.

#### *Funnel Method*

#### Apparatus

5. The funnel type viscosimeter shall be constructed as shown in Fig. 1.

#### Procedure

6. The tests shall be made at  $77 \pm 3.6$  F. ( $25 \pm 2$  C.). Both the sample of cement to be tested and the apparatus

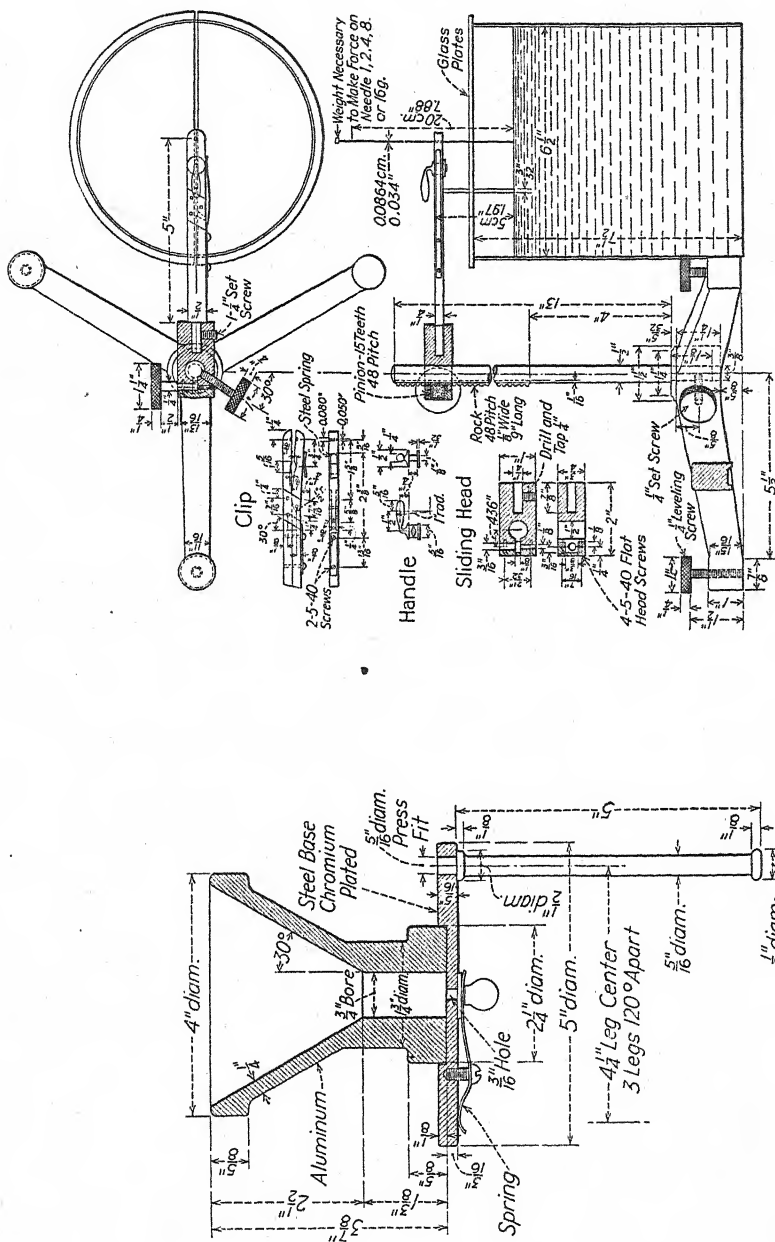


FIG. 1.—Funnel Type Viscosimeter.

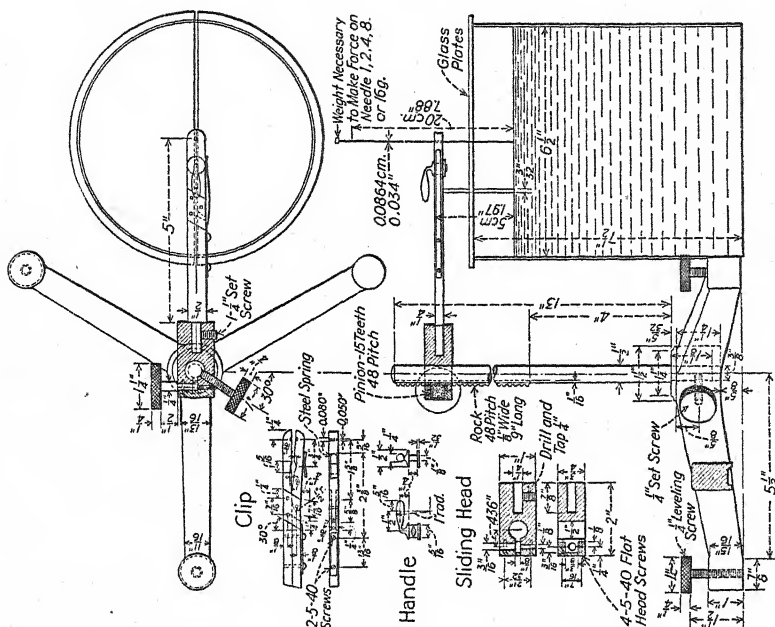


FIG. 2.—Falling Cylinder Viscosimeter.

shall be adjusted to this temperature before starting the test. The viscosimeter shall be adjusted so that the orifice is perpendicular to a smooth horizontal surface. With the orifice closed the viscosimeter shall be charged with 125 ml. of cement (Note). The orifice shall then be opened and the cement allowed to flow. The time of flow shall be measured by means of a stop watch.

NOTE.—In charging the viscosimeter, care must be exercised that no air is trapped in the straight portion below the funnel. The best precaution against trapping air is to pour the sample slowly into the side of the funnel until the straight portion is filled.

### Report

7. The viscosity shall be reported as the number of seconds required for the sample of cement to discharge until the liquid level is just at the bottom of the conical section of the funnel.

### *Falling Cylinder Method*

#### Apparatus

8. The falling cylinder viscosimeter shall be constructed as shown in Fig. 2. Each cylinder shall be marked with two circumferential lines, one line 5.00 cm. from the lower end, and the other line 15.00 cm. from the first line. The cylinders shall conform to the following requirements:

Cylinder	Diameter, in.	Weight, g.
No. 1.....	0.034	1.047
No. 2.....	0.034	2.047
No. 3.....	0.034	4.047
No. 4.....	0.034	8.047
No. 5.....	0.034	16.047
No. D.....	0.032	0.306

### Procedure

9. The temperature of the sample shall be adjusted to  $77 \pm 3.6$  F. ( $25 \pm 2$  C.). After attaching the clip to the ring stand, a 1-gal. can of cement shall be placed directly under the clip. The clip shall be adjusted to such a height that the lower end of the adjustment needle just touches the surface of the cement. In this position the first mark on the cylinder will be flush with the top of the clip. The surface of the cement shall be skimmed, the cylinder immediately positioned, and the control on the cylinder released while at the same time a stop watch shall be started to record the time. The needle shall be kept falling freely through the clip by moving the container as required. The test is completed when the second mark reaches the clip, corresponding to a drop of 15 cm. To remove the needle after a test, release the jaws of the clip by means of the trigger. This prevents cement running into the orifice.

### Report

10. The viscosity shall be reported as the number of seconds required for the specified needle to fall through the orifice in the clip from one marker to the other.

# *Standard Methods of*

## SAMPLING AND TESTING LATICES OF NATURAL RUBBER AND SYNTHETIC RUBBERS<sup>1</sup>



A.S.T.M. Designation: D 640 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 640; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods are intended for use, as required, in sampling and measuring variability in composition of normal and concentrated rubber latex and synthetic rubber latices.

NOTE.—Other determinations in addition to those described in these methods are frequently used in testing rubber latex but have not been included, pending further development.

### Sampling Latex in Drums

2. (a) In case the sampling operation takes place immediately after the drum is filled, no further mixing is required, but if the drum has stood, the top shall be removed and the contents stirred with a high-speed stirrer for 10 min.

(b) If closed-head drums are used, a procedure of rolling and up-ending the drums shall be employed; rolling alone is not sufficient. If there is a free air space in the drum, satisfactory mixing can be accomplished within a short time by this means. In the case of full drums

all of the latex in the closed-head drum shall be transferred to a larger vessel and mixed thoroughly before taking the sample.

(c) A 1-liter (32-oz.) dry, open bottle shall be attached to a 120-cm. (4-ft.) length of 0.625-cm. (0.25-in.) steel rod. The bottle thus attached to the rod shall be introduced into the drum and run down through the entire body of latex. The bottle shall then be withdrawn, and the contents emptied back into the drum. This initial filling minimizes the possibility of error from moisture that may be in the bottle. The bottle shall then be filled again in the same manner. It will be found advantageous to leave a small air space in the top of the bottle after the second filling in order to facilitate mixing before weighing out samples.

### Sampling Latex in Tank Cars

3. (a) In case the tank car has been freshly filled, no further mixing will be necessary. If the car has stood, the latex shall be mixed by means of a jet of air from a 1.25-cm. (0.5-in.) pipe inserted through the dome cover of the car and moved continuously throughout the body of the latex. In the case of

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.  
<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1941 to 1944, being revised in 1944.

normal latex, this operation shall be continued for 15 min., and in the case of creamed or centrifuged latex it shall be continued for about 45 min.

(b) When the contents of the car have been thoroughly mixed, a 1-liter (32-oz.) narrow-mouth sample bottle attached to a 300-cm. (10-ft.) length of steel rod shall be introduced into the car. The bottle shall be withdrawn and the contents emptied back into the car. This initial filling minimizes the possibility of error from moisture that may be in the bottle. Next, the bottle shall be forced quickly down through the body of the latex, and raised and lowered rapidly through the entire depth of the tank car several times. On account of the narrow mouth, there will be time to do this during the period of filling of the bottle. The bottle shall be withdrawn when it is completely filled. Just enough latex shall be poured out to leave a small air space in the top, and the bottle shall then be stoppered tightly.

#### Total Solids

4. (a) Weigh  $2.5 \pm 0.5$  g. of the latex to be tested into a tared, covered, flat-bottom glass weighing dish. The latex should be uniformly distributed over the bottom of the dish, during drying, over an area of approximately 32 sq. cm. (5 sq. in.). With the dish uncovered, dry the specimen in air for 16 hr. at 70 C. Cool the dried film to room temperature and weigh.

(b) Calculate the percentage of total solids as follows:

$$\text{Total solids, per cent} = \frac{\text{wt. of dried film}}{\text{wt. of latex sample}} \times 100$$

#### Dry Rubber Content

5. (a) Weigh a representative sample of not less than 20 g. of normal latex or 10 g. of concentrated latex into a porcelain evaporating dish, and add distilled water until the total solids content is

approximately 25 per cent. Add acetic acid (2.0 per cent), while stirring constantly, until the latex appears to be coagulated and more acid produces no effect. Place the dish on a steam bath for 30 min., then pour off the serum and replace it with distilled water. Remove the coagulum and pass it between the tightly closed rolls of a laundry wringer or similar device, wash it again with distilled water, and wring out. Repeat this process five times. Dry the resulting crepe to constant weight at 70 C.

(b) Calculate the dry rubber content as follows:

$$\text{Dry rubber content, per cent} = \frac{\text{wt. of dry coagulum}}{\text{wt. of sample}} \times 100$$

#### Preparation of Sample for Chemical Analysis

6. Spread the latex to be dried on a flat glass plate. Convenient amounts to use are 1 ml. of normal latex per 6.45 sq. cm. (1 sq. in.) of surface or 0.5 ml. of concentrated latex per 6.45 sq. cm. Place the plate in a current of warm air at approximately 90 F., and allow the latex to dry slowly to a transparent film.

NOTE.—This method of preparing a dried film of latex rubber is specified in order to obtain in a uniform manner a material that can be directly subjected to the procedures for determination of manganese, copper, and acetone extract as described in the Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297) of the American Society for Testing Materials.<sup>3</sup>

#### Report

7. The report shall include the following:

(1) Identification of sample and statement of the method of sampling used, and

(2) Results of all determinations made.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# *Standard Methods of*

## TESTING COMPRESSED ASBESTOS SHEET PACKING<sup>1</sup>



A.S.T.M. Designation: D 733 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 733; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods describe test procedures for evaluating the physical properties of the product known commercially as "compressed asbestos sheet packing" manufactured by bonding a large volume of asbestos fiber with a compound of natural or synthetic rubber or a mixture of both. In testing any particular type or grade of packing, appropriate procedures may be selected from those given.

### General Methods

2. (a) Except as otherwise specified in these methods for compressed asbestos sheet, the following methods of test of the American Society for Testing Materials, applicable in general to rubber products, shall be complied with as required and are hereby made a part of these methods:

- (1) *Tension Test*.—Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412).<sup>3</sup>

- (2) *Aging Test*.—Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573).<sup>3</sup>

- (3) *Immersion Test*. — Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (A.S.T.M. Designation: D 471).<sup>3</sup>

(b) In all tests described in these methods, the test specimens shall be conditioned in an oven at 100 C. for 1 hr. and allowed to cool to room temperature in a desiccator before proceeding with the tests.

(c) Test conditions indicated in these methods as "specified" shall be as prescribed in the contract or purchase specifications for the particular material being tested. In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular lot of material, the latter shall take precedence.

### Composition

3. Whenever composition require-

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1943 to 1946, being revised in 1943 and 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



ments are prescribed in purchase specifications, the conformity of the material to these requirements shall be supported by affidavits from the manufacturer rather than by chemical analysis (Note).

NOTE.—Present known methods of chemical analysis of this type of material do not have the degree of accuracy required for specification control tests.

### Sampling

4. One sample shall be taken from each 1000 lb. or fraction thereof of packing having any one thickness. This sample shall be of sufficient size to provide the necessary specimens for the tests required. In general, a sample 12-in. square will suffice. No samples shall be selected from material below  $\frac{1}{4}$  in. or above  $\frac{1}{2}$  in. in thickness. Material of thicknesses not included in this range shall be represented by samples of acceptable thickness which are guaranteed by the manufacturer to be of equivalent composition and cure.

### Measurement of Thickness

5. Thickness shall be determined in accordance with the procedure described in Section 8 (d) of A.S.T.M. Methods D 412, except that the thickness recorded shall be the average of not less than five separate measurements; the presser foot shall exert a force of  $9.0 \pm 0.1$  oz.; and the diameter of both presser foot and anvil shall be  $0.25 \pm 0.01$  in.

### Tensile Strength

6. The tensile strength shall be determined in accordance with Methods D 412<sup>3</sup> using specimens cut with die A, except that the testing machine shall have a jaw separation speed of 12 in. per min. Die B may be used as an alternate when the thickness of the sample is such that the breaking load for specimens cut with die A might be in excess of the capacity of the testing machine. Specimens shall not be buffed

prior to testing. The number of test specimens and method of calculating results shall be in accordance with Section 9 (a) of Methods D 412, except that three specimens shall be selected in each of two directions at right angles to each other, corresponding to the longitudinal and transverse directions of the calendar grain in the original sheet. Special care shall be employed in inserting specimens into the grips of the testing machine to insure that they are accurately aligned (Note). Results in each direction shall be calculated as the average of the three tests.

NOTE.—Errors as high as 50 per cent can result from imperfect alignment.

### Bending Test

7. A specimen of the packing 1 in. in width and of sufficient length to be grasped by the ends shall be bent 180 deg. around a rod of specified diameter with just enough force to hold the specimen in intimate contact with the rod. Cracking of the specimen where bent, as determined visually, shall be considered failure to pass the test. The test shall be applied to strips cut in both longitudinal and transverse directions unless otherwise specified.

### Compressibility

8. A specimen having a minimum area of 1 sq. in. either in the form of a square or circular disk shall be cut from the sample and placed on a hardened steel plate in any standard type of compression testing machine. A flat compression loading disk  $\frac{1}{2}$  in. in diameter made of hardened steel shall be placed in the center of the specimen on the opposite side from the plate. The specified load shall then be applied at a slow uniform rate and held for 30 sec. at which time the deflection shall be read by the use of suitable micrometers. The percentage

of compressibility shall be calculated as follows:

Compressibility, per cent =

$$\frac{\text{original thickness} - \text{compressed thickness}}{\text{original thickness}} \times 100$$

#### Accelerated Aging

9. The accelerated aging test shall be conducted by the oven method in accordance with Method D 573. The aging period shall be 96 hr. at 70 C. Extent of the aging shall be measured by the change in the results of the tensile strength, bending, and compressibility tests made in accordance with the procedures described in Sections 6, 7, and 8, when carried out before and after the aging period.

#### Immersion Tests

10. (a) The ability of the packing to withstand the effect of liquids shall be estimated from immersion tests in specified liquids for stated periods at the required temperatures. Two procedures are given designated as method A and method B. In method A, the test specimens are cut from the sample and immersed without any conditioning procedure. In method B, strips of the sample as required are conditioned according to the procedure described in Paragraph (c), prior to the cutting of the immersion test specimens. Method B shall be employed only when specified for high-temperature immersion tests on types of compressed asbestos sheet which are designed to develop their proper fluid resisting characteristics after application in service at elevated temperatures.

(b) *Method A.*—The material shall be tested in accordance with method A of Methods D 471, except that the thick-

ness of the specimen shall be that of the material and the specimens intended for determination of tensile strength shall be cut with die A of Methods D 412 in both the longitudinal and transverse directions of the sheet. The calculation of tensile strength after immersion shall be based on the original cross-section of the test specimen rather than on the swollen area.

(c) *Method B.*—Strips of packing 6 in. in length by 3 in. in width shall be cut from the sample in both the longitudinal and transverse directions as required. Each strip shall be placed between smooth surfaces in a compression device consisting of steel plates 6 by 6 by 1 in. provided with four bolts for drawing the plates together. The strips shall be compressed and held at 5 per cent less than the original thickness. The assembly shall be placed in a thermostatically controlled air oven for 16 hr. at the temperature at which the subsequent immersion period is to be conducted. At the end of this time the strips shall be removed from the conditioning apparatus and allowed to cool for 1 hr. in air at room temperature, after which the immersion test specimens shall be cut from the strips. These conditioned specimens shall then be tested in accordance with method A as described in Paragraph (b).

(d) The effect of immersion shall be judged by changes in thickness, weight, tensile strength, or compressibility made in accordance with the procedures described in these methods in Sections 5, 6, and 8, and in method A of Methods D 471, except that in the case of immersion tests in volatile liquids, it shall be judged only by changes in thickness or weight. The measurement of thickness shall be completed within 30 sec. after removal from the liquid.

### Corrosion Test

11. A rectangular specimen  $\frac{1}{2}$  by 2 in. cut from the sample shall be placed on a section of a specified metal and held in contact by a 1-lb. load. This assembly shall be immersed in a specified medium for 16 hr. at 90 C. A blank control test using neutral filter paper in place of the test specimen shall be made simultaneously. At the end of the immersion period the assemblies shall be removed from the bath and allowed to cool for 1 hr. Visible pitting in excess of that observed in the control test shall be regarded as evidence of corrosion.

### Retests and Rejection

12. Because of the nonhomogeneous nature of compressed asbestos sheet packing, any material which fails to meet the requirements in one or more of

the specified tests, may be resampled and retested. For this purpose, one additional sample shall be selected and specimens prepared and tested according to the procedure for the test in which the original specimen failed to meet the requirements. In the event of failure of this retest, the packing represented by the sample tested shall be rejected.

### Report

13. The report shall include the following:

- (1) Description of the sample including lot identification, type, and thickness of sheet,
- (2) Statement of methods used, specified test conditions, and results obtained,
- (3) All observed and recorded data, and
- (4) Date of manufacture of the material, if known, and date of test.

## *Standard Method of Test for* DEGREE OF STAINING OF PAPER BY ALKALI<sup>1</sup>



A.S.T.M. Designation: D 723 - 45

ADOPTED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 723; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test for determining the degree of staining of paper by alkali is applicable to undyed papers. It can be used with hard-sized (rosin) papers by first removing the major part of the sizing with ether or methanol. It is not recommended for use with pulp.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Nessler Tubes*.—A set of six Nessler tubes made with thin, colorless glass, 1 mm. in thickness, having a diameter of 29 to 30 mm., and with the 50-ml. graduation mark about 90 mm. from the bottom outside.

(b) *Precision Pipette*. — A precision pipette of 1-ml. capacity, graduated to 0.01 ml.

### Reagents

#### 3. (a) *Potassium Dichromate Solu-*

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

For further information on this method the following reference may be consulted:

T. Linsey Crossley, "A Quantitative Test for Soap Wrap Paper," *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Series XX (1937).

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1943 to 1945.

*tion*.—Dissolve 0.25 g. of  $K_2Cr_2O_7$  in a small amount of water and dilute to 1 liter.

(b) *Congo Red Solution*.—Dissolve 0.5 g. of congo red<sup>3</sup> in water, and dilute to 1 liter.

(c) *Sodium Hydroxide Solution (1 N)*.

### Reference Standards

4. To 50 ml. of the potassium dichromate solution add 0.1 ml. of the congo red solution and mix well. Place different amounts of this mixed solution, such as 0.2, 0.5, 1.0, and 2.0 ml. each, in separate Nessler tubes, dilute to the 50-ml. mark with water, and mix.

NOTE: *Caution*.—As potassium dichromate slowly destroys the congo red color, the solution should not be mixed until immediately before use.

### Test Specimen

5. The test specimen shall consist of 3.00 g. of the paper torn into pieces  $\frac{1}{4}$  to  $\frac{1}{2}$  in. square. With vegetable parchment a finer subdivision of the sample is advisable.

<sup>3</sup> Eastman Kodak Co. Congo Red E.K. 770 is recommended.

### Procedure

6. (a) Place the test specimen in a 250-ml. Erlenmeyer flask, add 50 ml. of hot water and boil for 5 min. Decant the liquid into a 100-ml. flask or graduated cylinder. Add 50 ml. of hot water to the paper in the flask and boil again for 5 min. Decant the second liquid into the first and dilute to 100 ml., using the water for dilution to wash the paper in the flask.

(b) Add 25 ml. of 1 *N* NaOH to the combined liquids and, after standing for at least 5 min., filter, using a fast filter paper if the insoluble matter is flocculent, or a close filter if it appears cloudy.

(c) Place 50 ml. of the filtered alkaline solution from the paper in a Nessler tube and compare with the reference standards. The solutions are best compared

by holding the tubes containing them over white paper, but not resting on it, and looking down through the solutions. If none of the reference standards matches the solution, prepare reference standards of other strengths and compare them with the solution. Exact matches of tint are not always possible, but distinction in intensity is readily seen.

### Report

7. Report the number of milliliters of potassium dichromate - congo red solution required to match the tint of the alkaline extract of paper as the *alkali-staining number*. Express the results to one decimal place.

NOTE.—It has been found that papers showing an alkali staining number of more than 3 will give stain with NaOH (1 per cent) by the drop test.

## *Standard Method of Test for*

### ASH CONTENT OF PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 586 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 586; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. This method of test is intended for use in determining the ash content of paper and paper products, which is defined as the residue after complete combustion of the paper. The mineral content of paper may consist of: (1) various residues from chemicals used in its manufacture, (2) metallic matter from piping and machinery, and (3) filling, coating, and pigmenting materials. Generally, if the ash content does not exceed about 2 per cent, no filling, coating, or pigmenting material has been used; but this is not always the case, as pigments of high opacity or coloring power are sometimes used in very small amounts. When filling and coating materials are present that do not change much on ignition, such as barium sulfate and certain titanium pigments, the ash is an approximate measure of the amount present.

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Crucible*.—A crucible, made of material such as platinum, alundum, porcelain, or silica, that does not change in weight under the ignition conditions, and having a tightly fitting lid.

(b) *Heat Source*.—An electric muffle furnace with an operating temperature of approximately 925 C. (1700 F.) is recommended, but a gas burner yielding a similar temperature is also suitable.

#### Test Specimen

3. The test specimen shall consist of small pieces of paper so selected as to be representative of the sample. Its total weight shall be not less than 1 g.

#### Procedure

4. (a) Dry the specimen to constant weight at  $105 \pm 2$  C. and weigh to the nearest 1 mg. This may be done with sufficient accuracy for the purpose in the ignited and weighed crucible used for the ashing of the specimen.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

(b) Ignite the dried specimen in the crucible, which, together with the cover, has previously been ignited and weighed. To avoid loss of small particles, the crucible shall be covered during the initial ignition of the specimen, which shall be done at low temperature. The temperature shall then be gradually raised to a maximum of approximately 925 C. (1700 F.). After this temperature is reached, the lid of the crucible may be slid to one side until the combustion is complete. Care shall be taken at all times to protect the contents of the crucible from air drafts. When the specimen is completely burned, as indicated by absence of black particles, remove the covered crucible to a desiccator, and allow it to remain until its temperature is in equilibrium with that of the surrounding atmosphere. Weigh the crucible and contents to the nearest 0.1 mg. Repeat the ignition

and weighing until the weight is constant.

#### Calculation and Report

5. (a) *Calculation.*—The percentage of ash shall be calculated on the basis of the weight of the specimen dried to constant weight at  $105 \pm 2$  C.

(b) *Report.*—The percentage of ash shall be reported as the average of at least two determinations. Ash shall be reported to the nearest 0.05 per cent for papers containing 5 per cent ash or less, to the nearest 0.1 per cent for papers containing 5 to 10 per cent ash, and to the nearest 0.2 per cent for papers containing more than 10 per cent ash.

#### Reproducibility of Results

6. Results of duplicate determinations should agree within the following:

Ash Content	Permissible Difference
5 per cent or less.....	0.1
Over 5 to 10 per cent.....	0.15
Over 10 per cent.....	0.2

## *Standard Method of Test for* BASIS WEIGHT OF PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 646 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 646; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers the procedure for determining the basis weight of large and small pieces of paper. Factors for conversion of basis weight from one commercial size of paper to another are given in the Appendix.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Balance*.—A balance having a sensitivity of not less than 0.25 per cent of the load applied and so graduated that readings of this degree of accuracy can be made. The balance should preferably be a specially constructed sheet-weighing device indicating the equivalent weight of a 500-sheet and also a 480-sheet ream in pounds when a specimen consisting of one sheet of the designated size is weighed. The balance shall be protected from air currents while weighing.

(b) *Measuring Device*.—A device capable of measuring the size of the speci-

men to an accuracy within 0.25 per cent of the smallest dimension to be measured.

(c) *Paper Cutter*.—A template for cutting the paper to size. When a template is used for preparing the sheets, it is recommended that the paper be cut to exact size with a sharp knife. A paper cutter having an attachment for ensuring parallelism of the opposite edges is also recommended.

### Calibration of Balance

3. The balance shall be level at all times and care shall be taken that the level is not disturbed. The balance shall be calibrated after leveling by applying accurate weights with increasing and decreasing loads. If the level of the balance is not disturbed, the frequency of calibration depends entirely upon the condition and frequency of use and, therefore, becomes largely a matter of judgment.

### Sampling

4. The material shall be sampled in accordance with the Standard Method of Sampling Paper and Paper Products

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1944, being revised in 1942.



(A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>3</sup>

### Test Specimens

5. The test specimens shall consist, whenever possible, of at least 10 sheets each approximately 100 sq. in. (645 sq. cm.) in area taken from the sample obtained as prescribed in Section 4.

### Test Conditions

6. All test specimens shall be brought to a standard condition, prior to testing, in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials.<sup>3</sup> All tests shall be made under the standard atmospheric conditions.

### Procedure

7. The area of the test specimen shall be determined to the nearest 0.25 per cent of its total area. The weight of the specimen shall be determined to the nearest 0.25 per cent of its total weight.

If weighed in grams, the weight of 10 sheets multiplied by 1.102 gives the equivalent weight in pounds of 500 sheets of the same area.

### Report

8. (a) The report shall include at least two of the following items:

(1) Equivalent ream weight in pounds for a ream consisting of 500 sheets 25 by 40 in. (635 by 1016 mm.) in size,

(2) Weight in grams per square meter of paper, and

(3) Equivalent weight for the ream size commonly used by the paper industry for the particular type of paper.

(b) The weight shall be reported to three significant figures. If less than 1000 sq. in. (0.6 sq. m.) of paper is used for the test, the area tested shall be reported.

### Reproducibility of Results

9. Duplicate determinations made on the same sample shall agree within 2 per cent.

## APPENDIX

A1. The information given below will be of assistance in calculating the weight of paper and the conversion of customary trade-sizes 500-sheet reams to and from the weight in pounds of size 25 by 40 in., and to the weight in grams per square meter.

A2. When specimen sheets are weighed on a balance in grams or on a paper scale, the weight of trade size 25 by 40 in., 500-sheet ream is calculated as follows:

$$P = \frac{1102g}{abn} \quad \text{or} \quad \frac{7111g}{yzn}$$

$$P = \frac{1000p}{abn} \quad \text{or} \quad \frac{6451p}{yzn}$$

$$G = \frac{1550g}{abn} \quad \text{or} \quad \frac{10,000g}{yzn}$$

$$G = \frac{1406p}{abn} \quad \text{or} \quad \frac{9070p}{yzn}$$

where:

$P$  = weight in pounds of trade size 25 by 40 in., 500-sheet ream,

$G$  = weight in grams per square meter of trade size 25 by 40 in., 500-sheet ream,

$a$  = length of specimen in inches, or

$y$  = length of specimen in centimeters,

$b$  = width of specimen in inches, or

$z$  = width of specimen in centimeters,

$g$  = weight in grams of sheets weighed, or

$p$  = indicated weight on scale for 500 sheet ream, and

$n$  = number of sheets weighed.

A3. Conversion factors for converting the weights of some customary trade sizes of 500-sheet reams,  $t$ , to and from the weight in pounds of 25 by 40 in., 500-sheet reams,  $s$ , and in grams per square meter,  $m$  are given in Table I,

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE I.—CONVERSION FACTORS FOR TRADE SIZES OF PAPER.<sup>4</sup>

Kind of Paper	Trade Size, 500-sheet Ream, in. <sup>a</sup>	Conversion Factors <sup>b</sup>			
		A (s to t)	B (m to t)	C (t to s)	D (t to m)
Writing and printing (Demy size).....	17 by 22	0.374	0.266	2.674	3.760
Blotting.....	17½ by 22½	0.394	0.280	2.540	3.571
Cover.....	19 by 24	0.456	0.324	2.193	3.083
Tissue (double crown size).....	20 by 26	0.520	0.370	1.923	2.704
Cardboard.....	20 by 30	0.600	0.427	1.667	2.343
Bristol and tag.....	22 by 28	0.616	0.438	1.623	2.282
News and wrapping.....	22½ by 28½	0.641	0.456	1.559	2.195
Book.....	24 by 36	0.864	0.614	1.157	1.627
T.A.P.P.I. standard size.....	25 by 38	0.950	0.675	1.053	1.480
Metric size.....	25 by 40 1 sq.m.	1.000 1.406	0.711 1.000	1.000 0.711	1.406 1.000

<sup>a</sup> If the given trade size is in 480-sheet reams, then the factors A and B shall be multiplied by 48/50 (or 0.960), and C and D by 50/48 (or 1.042) for conversion.

<sup>b</sup> These conversion factors, except for the metric size, are obtained as follows:

A = 1/1000 area of trade size sheet in square inches

B = 0.711 A

C = 1/A

D = 1.406/A

A4. The following examples illustrate the use of the conversion factors in practical calculations:

(a) The weight in pounds of 25 by 40-500 multiplied by A is equivalent to the weight in pounds of a given trade size, for example:

$$100 \text{ lb. } 25 \text{ by } 40-500 \times 0.600 = 60.0 \text{ lb. } 20 \text{ by } 30-500$$

(b) The weight in grams per square meter multiplied by B is equivalent to the weight in pounds of a given trade size, for example:

$$60 \text{ g. per sq. m.} \times 0.711 = 42.7 \text{ lb. } 25 \text{ by } 40-500$$

(c) The weight in pounds of a given trade size multiplied by C is equivalent to weight in pounds of 25 by 40-500, for example:

$$32 \text{ lb. } 24 \text{ by } 36-500 \times 1.157 = 37.0 \text{ lb. } 25 \text{ by } 40-500$$

(d) The weight in pounds of a given trade size multiplied by D is equivalent to the weight in grams per meter, for example:

$$50 \text{ lb. } 25 \text{ by } 38-500 \times 1.480 = 74 \text{ g. per sq. m.}$$

<sup>4</sup> Editorially revised in November, 1945.

A5. For convenience in converting the weight of a ream of 500 sheets 24 by 36 in. in size to the weight of a ream of 500 sheets of any other of the accepted trade customs, multiplier factors are given in Table II.

TABLE II.—CONVERSION FACTORS FOR WEIGHTS OF REAMS.<sup>4</sup>

Trade Custom Size, 500-sheet Ream, in.	Conversion Factor
17 by 22.....	0.4329
19 by 24.....	0.5278
20 by 26.....	0.6018
20 by 30.....	0.6944
22 by 28.....	0.7129
22.5 by 28.5.....	0.7422
24 by 36.....	1.000
25 by 38.....	1.0995

A6. (a) In case the available sample is smaller than the required size as prescribed in Paragraph A5, the following formula should be used for obtaining weight in the desired ream size. The paper should be cut to accurate dimensions and weighed on an analytical balance.

$$T = \frac{WS}{a}$$

where:

T = weight of trade size desired,

W = weight of sample in pounds,

S = area of trade size in square inches, and

a = area of sheet in square inches.

(b) When it is required to find the linear feet in a roll of paper having given the ream weight, the width of the roll, etc., the length of the roll may be calculated as follows:

$$L = \frac{Ra}{12WD}$$

where:

L = length of roll in feet,

R = weight of roll in pounds,

a = area of ream in square inches,

W = weight of ream in pounds, and

D = width of roll in feet.

## Standard Method of Test for BURSTING STRENGTH OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 774 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 774; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers the procedure for measuring the bursting strength of paper and paper products having a bursting strength of not over 200 psi. and occurring as single or laminated flat sheets not over 0.025 in. in thickness.<sup>3</sup> This method is not intended to be used for testing corrugated box board.

### Definition

2. Bursting strength is defined for this method of test as the hydrostatic pressure required to produce rupture of a circular area of the material under

test 1.2 in. in diameter, when applied at a controlled, increasing rate, as described in Section 6. During the test, the specimen must be free to bulge under influence of the increasing pressure, but the periphery of the test area must be rigidly fixed so that it does not move while the pressure is being applied.

### Apparatus

3. The testing machine shall conform to the following requirements:

(a) *Clamps*.—A means for firmly clamping the test specimen without slippage during the test between two annular, plane, unpolished (matte) surfaces, which may have fine, concentric tool marks not over 0.002 in. in depth for the purpose. The upper clamping surface or the clamping ring shall have a circular opening  $1.200 \pm 0.001$  in. in diameter. The circular edge of the opening which is in contact with the paper during the test shall be relieved of sharpness, but not rounded off enough to alter significantly the diameter of the opening. The lower clamping surface or the diaphragm plate

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

<sup>3</sup> For further information on this method the following references should be consulted:

F. T. Carson, and F. V. Worthington, "A Critical Study of the Bursting Strength Test for Paper," *Journal of Research*, Nat. Bureau Standards, Vol. 6, p. 339 (1931) (*Research Paper RP 273*).

F. T. Carson, "Some Notes on the Revision of Methods for Measuring the Strength of Paper," *Paper Trade Journal*, Vol. 102, p. TS 263 (1936).

James d'A. Clark, "Determining the Bursting Strength of Paper," *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Series IV, pp. 367-370 (1932).

C. V. Oliver, "Variability in Test Results," *Proceedings*, Tech. Section, Paper Manufacturers' Assn., Vol. 11, p. 53 (1930).

C. F. Underhay, "Bursting Tester Standardization," *Proceedings*, Tech. Section, Paper Manufacturers' Assn., Vol. 11, Part 2, p. 247 (1931).

shall be 0.125 in. in thickness and shall have an opening  $1.25 \pm 0.01$  in. (1.24 in. recommended) in diameter. Its edge which in use is in contact with the rubber diaphragm shall be rounded off sufficiently to prevent it cutting the rubber when pressure is applied. When testing a specimen, the annular openings of the two clamping plates shall be co-axial.

(b) *Rubber Diaphragm*.—A rubber diaphragm, of pure gum rubber, free from mineral loading material and 0.033 to 0.035 in. in thickness, clamped between the lower clamping plate and the rest of the apparatus, so that before the diaphragm is stretched by pressure underneath it, the center of its upper surface is below the plane of the clamping surface. A pressure of 0.5 psi. shall be capable of distending the diaphragm through the aperture of the clamping plate to a height of at least 0.125 in. above the top surface of the plate. It is recommended that this diaphragm be renewed monthly.

(c) *Motor*.—A means of applying controlled, increasing hydraulic pressure to the underside of the diaphragm until the specimen bursts. This pressure shall be generated by a motor-driven piston forcing a liquid (usually glycerine) into the pressure chamber of the apparatus at the rate of 75 ml. (about 6 cu. in.) per min. (Note).

NOTE.—This rate is attained in the hand-driven instrument by turning the hand wheel 120 rpm. Since the testing rate of both types of instruments may be changed by air trapped in them, care should be used to exclude air when the diaphragms or gages are changed.

(d) *Reading Pressure Gage*.—A maximum reading pressure gage, with a dial preferably 5 or more inches in diameter for indicating bursting pressures in pounds per square inch with the following accuracy:

Pressure, psi.	Accuracy, psi.
10 or less.....	0.25
11 to 45, incl.....	0.5
46 to 100, incl.....	1.0
101 to 200, incl.....	2.0

### Calibration of Apparatus

4. The pressure gage shall be calibrated, while inclined at the same angle at which it is used during tests, by means of a dead-weight gage tester of the piston type, or by means of a column of mercury. During calibration, the pressure shall be applied as specified in Section 3 (c) (Note). Gages in frequent use should be calibrated monthly. If a gage is accidentally used "over capacity," it should be calibrated before using again.

NOTE 1.—The rate of applying the pressure can be regulated by means of the valve on the dead-weight tester. The regulation is facilitated by providing the valve with a liner stop. Unless the speed of building up the pressure in the testing instrument is too high, it is probably not very important to take account of it in the calibration.

### Test Specimens

5. The test specimens shall be the equivalent of a sufficient number (at least 10) of sheets of paper each at least 2.5 by 2.5 in., to allow for making the number of tests required in Section 6. They shall be representative of the sample obtained according to the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585).<sup>4</sup>

### Procedure

6. (a) The test shall be made in an atmosphere conditioned according to the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685),<sup>4</sup> and after the specimens have reached equilibrium

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

in the specified atmosphere. The gage used for measuring the bursting pressure shall be such that the individual readings will be not less than 25 and not more than 75 per cent of the total capacity of the gage. Therefore, unless the approximate strength of the paper under test is known, preliminary tests should be made to determine the required capacity of the gage.

(b) The specimen shall be clamped securely in position, and the hydrostatic pressure applied as specified in Section 3(c) until the specimen ruptures. The maximum indication registered by the pressure gage shall be recorded. The unclamped margin of the specimen shall be watched carefully for any movement. If slippage is indicated, the test value shall be discarded and the clamping pressure increased for the remainder of the tests. At least ten tests shall be made using as many of the sheets comprising the test specimen as possible. In one half of the tests, pressure shall be applied to the wire side of the paper, and, in the other half, to the felt side.

(c) No tests shall be made in water marks, or areas containing creases, imperfections, or visible damage. After each test the indicator needle of the gage shall be returned gently to zero.

### Report

7. The report shall include the following:

(a) Test results, corrected for gage error and reported to three significant figures, as maximum, minimum, and average bursting strength in pounds per square inch,

(b) The range of the scale of the pressure gage used, and

(c) The number of tests made.

### Reproducibility

8. Results of tests made on different samples from the same shipment, or on different apparatus should be expected to agree within 5 per cent, except for tissues or other weak papers with a breaking strength of 5 psi. or less.

## *Standard Method of Test for* CASEIN IN PAPER (QUALITATIVE)<sup>1</sup>



A.S.T.M. Designation: D 587 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 587; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method covers a qualitative test for casein in paper. It is applicable only to papers in which the amount of casein is relatively large, for example, mineral-coated papers in which casein is commonly used as the binder. It is not applicable to papers containing such small amounts of casein as may be used in the beater furnish as a constituent of the engine sizing, etc.

### Reagents

2. (a) *Sodium Hydroxide Solution* (1 per cent).

(b) *Nitric Acid* (sp. gr. 1.42).

(c) *Millon's Reagent*.—Dissolve 20 g. of pure mercury in 40 g. of c.p.  $\text{HNO}_3$  (sp. gr. 1.42) and dilute to 180 ml. with distilled water.

### Test Specimen

3. The specimen shall consist of about 0.5 g. of paper, cut into small pieces, and so selected as to be representative of the sample.

### Procedure

4. Boil the specimen several minutes in a test tube with 10 ml. of NaOH (1 per cent). (The NaOH is required to dissolve casein that has been hardened by formaldehyde or other agent.) Filter off the aqueous extract, cool to room temperature, add a suitable indicator, such as phenolphthalein, and exactly neutralize with  $\text{HNO}_3$  (sp. gr. 1.42). Add several milliliters of the Millon's reagent. Upon heating, the presence of casein is indicated by the development of a red coloration (Note).

NOTE.—This reaction is dependent on the presence of tyrosine which occurs in casein to the extent of approximately 5 per cent, but has been reported in only rare instances as occurring in animal glue and gelatin, and then only in doubtful traces.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

## Standard Method of Test for ALPHA-, BETA-, AND GAMMA-CELLULOSE IN PAPER.<sup>1</sup>



A.S.T.M. Designation: D 588 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 588; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method of test covers the procedures for determining alpha-, beta-, and gamma-cellulose, the three fractions into which cellulose can be separated analytically, as follows:

*Alpha-Cellulose.*—Alpha-cellulose is taken as the fraction that can be filtered out of a mixture consisting of the fibrous material and sodium hydroxide solution (7.3 per cent) of maximum dissolving power, after the fibers have previously been swelled with sodium hydroxide solution (17.5 per cent). After separation, the alpha-cellulose is determined either by drying and weighing, or volumetrically by oxidation with potassium dichromate. Both methods for alpha-cellulose are capable of the same reproducibility and give practically the same values. In the volumetric method, no moisture or ash determinations are made and a much smaller sample is used, resulting in a shorter and more rapid procedure.

*Beta - Cellulose.*—Beta - cellulose is taken as the fraction that precipitates at room temperature (15 to 35 C.) after the filtrate has been acidified, and is determined by the volumetric method.

*Gamma-Cellulose.*—Gamma-cellulose is taken as the fraction that remains in solution after removing beta-cellulose, and is determined by the volumetric method.

(b) The method of separating the alpha-cellulose from the other two fractions is intended primarily for papers made from rags or chemical wood fibers. If information is desired as to the applicability of the method to papers containing large amounts of lignin, such as newsprint, reference should be made to the original literature<sup>3</sup> for details of technique.

### Apparatus

2. The apparatus shall consist of the following:

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

<sup>3</sup> H. F. Launer, "Simplified Volumetric Determination of Alpha-, Beta-, and Gamma-Cellulose in Pulps and Papers," *Journal of Research, Nat. Bureau Standards*, Vol. 18, No. 3, p. 333 (1937) (*Research Paper 979*); also H. F. Launer, "Volumetric Determination of Alpha-, Beta-, and Gamma-Cellulose in Pulps and in Papers Containing Sizing, Filler, and Other Materials," *Journal of Research, Nat. Bureau Standards*, Vol. 20, No. 1, p. 87 (1938) (*Research Paper 1068*).

(a) *Grinder*.—A Koerner<sup>4</sup> or equivalent type grinder that will completely disintegrate the paper without heating or contaminating it.

(b) *Mixer*.—A device with which uniform mixture of the ground material can be secured. A mixer is easily constructed by fitting a crock or bell jar, of approximately equal diameter and depth, with a wooden lid through which runs the shaft of an electric fan, vanes being properly placed to produce turbulence. Mixing for 20 to 40 sec. at full speed normally suffices; longer periods result in separation of the light material from the heavy.

(c) *Bath*.—A water bath that can be maintained at  $20 \pm 0.1$  C.

(d) *Electrometric Titration Apparatus*.—For the estimation of dichromate, an indicator may be used as described in Section 3 (e), but for rapid, accurate analysis, an electrometric apparatus is recommended. Any ordinary potentiometric circuit with a platinum wire electrode and crude calomel half cell is suitable. More simply, a nichrome wire may be substituted for the calomel half cell. The nichrome wire is prepared for use by heating momentarily to bright redness, and then scraping the surface clean with a knife. The potentiometric arrangement may consist of a galvanometer with a sensitivity of 0.5 to 1 microampere per millimeter scale division, a dry cell, and a sliding-contact rheostat having a total resistance of approximately 400 ohms. Smaller rheostats will suffice but they drain the dry cell more quickly. The simplest form of the apparatus is shown in Fig. 1. The large deflection at the end point is unmistakable from possible slow creeping during the titration. The galvanometer

is adjusted to zero by varying the resistance at the beginning of the titration.

### Reagents

3. (a) *Sodium Hydroxide Solution* (17.5 per cent, 5.24 N).—Allow a 50 per cent solution of c.p. NaOH to stand about 1 week in a stoppered vessel to permit settling of  $\text{Na}_2\text{CO}_3$ . Draw off 2.00 ml. of the supernatant liquid with a pipette, add about 50 ml. of distilled

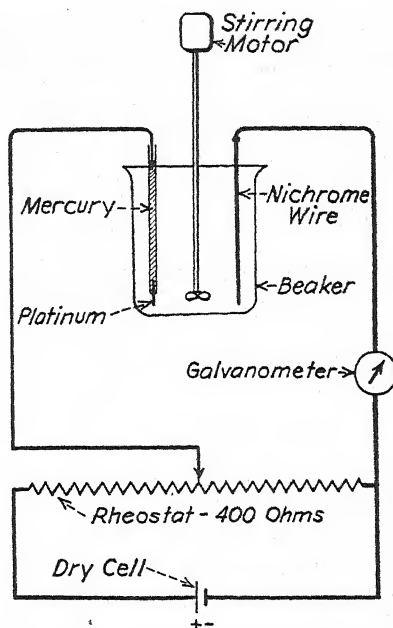


FIG. 1.—Electrometric Titration Apparatus.

water and 1 ml. of 1.5 N  $\text{BaCl}_2$  to lessen the effect of  $\text{CO}_2$  on the end point, and titrate with standard 1 N HCl, using phenolphthalein as indicator. Knowing the approximate normality of the concentrated NaOH, dilute it with distilled water to  $5.24 \pm 0.05$  N, checking the diluted NaOH by titrating 10.00 ml. of it as before, and diluting further, if necessary, to obtain the normality specified, which will correspond to  $17.5 \pm 0.2$  per cent.

<sup>4</sup> The Koerner type of grinder is described in a paper by J. O. Burton and R. H. Rasch on "The Determination of the Alpha-Cellulose Content and Copper Number of Paper," *Journal of Research*, Nat. Bureau Standards, Vol. 6, No. 4, p. 603 (1931) (Research Paper 295).



(b) *Potassium Dichromate Solution*.—Dissolve 90.0 g. of c.p., oven-dry (100 to 105 C.)  $K_2Cr_2O_7$  in hot water (70 to 90 C.), and dilute to 1 liter after allowing the solution to cool.

(c) *Ferrous Ammonium Sulfate Solution*.—Dissolve 195 g. of c.p.  $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$  in water containing 10 ml. of  $H_2SO_4$  (sp. gr. 1.84) and dilute to 1 liter. If the solution is kept out of contact with oxygen, by means of a slow, continuous stream of hydrogen, for example, its strength will remain quite constant. The amount of hydrogen thus used is about one fifth of a 200-cu. ft. cylinder per year. This precaution is not necessary, but reduces the frequency with which the potassium dichromate - ferrous ammonium sulfate ratio must be determined from once each day to two or three times a month.

(d) *Acetic Acid Solution for Gravimetric Procedure*.—Prepare a solution of acetic acid approximately 10 per cent by weight.

(e) *Dichromate Indicator*.—If electro-metric apparatus (Section 2 (d)) is not available, dissolve 0.3 g. of barium diphenylamine sulfonate and 0.5 g. of  $Na_2SO_4$  in 100 ml. of water. Use 5 to 10 drops in a solution sufficiently dilute for good observation of the end point, which is shown by a change from red to green. As an alternative, the outside indicator,  $K_3Fe(CN)_6$  solution, may be placed in drops upon white porcelain. The end point occurs when a drop of the solution being titrated turns a drop of the indicator blue.

(f) *Barium Chloride (1.5 N)*.—Dissolve 37 g. of  $BaCl_2 \cdot 2H_2O$  in water and dilute to 100 ml.

(g) *Sulfuric Acid (24 N)*.—Cautiously add three volumes of c.p.  $H_2SO_4$  (sp. gr. 1.84) to two volumes of water in a heat-resistant flask, cool by tap water, and add the acid in small portions, shak-

ing after each addition. The acid will then be approximately 72 per cent by weight.

(h) *Sulfuric Acid (6 N)*.—Dilute 1 volume of the 24 N  $H_2SO_4$  (Paragraph (g)) to 3.5 volumes in a graduated cylinder. After mixing, standardize against the 5.24 N NaOH (Paragraph (a)) and then dilute to  $6.0 \pm 0.1$  N.

### Test Specimen

4. The test specimen shall be cut from the sample in such a way as to be thoroughly representative. It shall be reduced to cotton-like form in the disintegrator and then thoroughly mixed. If the paper is mineral-coated, the coating shall be removed before grinding.

### ALPHA-CELLULOSE

#### *Volumetric Method<sup>3</sup>*

### Procedure

5. (a) Perform all operations (except the weighing described in Paragraph (b)) and keep all liquids, as nearly as possible, at  $20 \pm 0.1$  C.

(b) Weigh  $0.3 \pm 0.01$  g. of the specimen in a 100-ml. beaker. Add 20.0 ml. of NaOH (17.5 per cent), macerate until the fibers are uniformly wet and dispersed, and let stand 10 min. from the time of addition of NaOH. Then add 33 ml. of water, stir the mixture thoroughly, and let stand 1 hr. longer, stirring once during the interval. After stirring once more, pour about 5 ml. of the unsettled mixture on an 80-mesh copper or brass wire screen fitted into a Gooch crucible. The crucible and ring are supported by a funnel fitted into the neck of a 100-ml. volumetric flask with a rubber stopper through which passes a glass tube for suction. Form a mat with gentle suction (pressure differential, 10 to 20 mm. of mercury). Avoid excessive packing of the fibers, as this retards filtering. It may be necessary

to refilter the first filtrate, but loss of small amounts of alpha-cellulose to the filtrate does not affect the results appreciably. Pour the remainder of the mixture on the mat and, before the last of the liquid has run through, wash the beaker and the mat with 35 ml. of water.

(c) Moisten the residue of alpha-cellulose with water and remove it from the crucible. Place the crucible upright in a 400-ml. beaker, fill it with 25 ml. of 24 *N* H<sub>2</sub>SO<sub>4</sub> at room temperature, and rinse it after a few minutes with 50 ml. more of the acid. Disintegrate the alpha-cellulose pad in the acid, using a stirring rod. Add to the alpha-cellulose solution, with a pipette, 25.00 ml. of the K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, and heat at 140 to 150 C. for 10 min. Bubble air in a fine stream through the solution to prevent bumping, and keep the beaker covered with a watch glass, notched to permit entrance of a thermometer and the bubbling tube.

(d) After the solution has cooled to 130 C., add 50 ml. of water, rinse the thermometer, etc., and cool the solution to 60 C. or lower. Titrate the remaining K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> with the ferrous ammonium sulfate solution.

(e) Pipette exactly half of the filtrate from the alpha-cellulose, after all fibers present have settled, into a 400-ml. beaker containing 5.0 ml. of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>. If the paper contains oxidizable fillers such as ZnS pigment or CaSO<sub>3</sub>, filter the filtrate once through a thick pad of asbestos in a Gooch crucible before taking the portion for analysis. (Such fillers remaining with the alpha-cellulose may cause some error, but this is usually slight.) Cautiously, while stirring constantly, pour 50 ml. of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) down the side of the beaker containing the portion of the filtrate for analysis, then heat and titrate as described previously for the alpha-cellulose solution.

### Calculations

6. (a) Calculate the percentage of alpha-cellulose from the following Eqs. 1, 2, and 3, except that when resin, starch, or glue are present, corrections shall be made in accordance with Paragraph (b).

$$A = 25 - (v_1 \times r) \dots \dots \dots (1)$$

$$B = 2 \times (5 - v_2 \times r) \dots \dots \dots (2)$$

$$\text{Alpha-cellulose, per cent} = \frac{A}{A + B} \times 100 \dots (3)$$

where:

*A* = milliliters of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> required to oxidize the alpha-cellulose,

*B* = milliliters of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> required to oxidize the filtrate,

*v*<sub>1</sub> and *v*<sub>2</sub> = milliliters of ferrous ammonium sulfate required for titration of the K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> remaining after oxidation of the alpha-cellulose and filtrate, respectively, and

*r* = volume of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> equivalent to 1 ml. of ferrous ammonium sulfate solution (determined frequently by titrating 5 ml. of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in 100 ml. of diluted H<sub>2</sub>SO<sub>4</sub> (1:1)).

(b) If resin, starch, or glue are present, the K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> volumes shall be corrected as follows before calculating the percentage of alpha-cellulose: The amounts of sizing materials remaining with the alpha-cellulose are taken as 0.25 per cent glue, 0.2 per cent starch, and 0.2 per cent resin, based on the dry weight of the sample (Note 1). Convert these values into weights, and then into milliliters of the K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution by dividing each weight by the corresponding factors, which are 0.0154 g. per

ml. for glue, 0.0129 g. per ml. for starch, and 0.0066 g. per ml. for resin. Subtract the resulting volumes in milliliters of the  $K_2Cr_2O_7$  solution from the volumes of  $K_2Cr_2O_7$  consumed by the alpha fraction and by the beta-plus-gamma fractions, and substitute the corrected values in Eqs. 1 and 2 (Note 2).

NOTE 1.—These are average values but actual amounts have been found to vary not more than 0.1 per cent from them, irrespective of the content of glue, starch, or resin in the papers. For example, if a paper were found to contain 3.4 per cent glue, 0.7 per cent starch, and 1.1 per cent resin; then the sizing materials remaining in the beta-plus-gamma portion would be 3.15, 0.5, and 0.9 per cent, respectively.

NOTE 2.—The original literature should be consulted if the analyst is in doubt as to the further details of these calculations.

#### *Gravimetric Method (Alternative)*

##### **Procedure**

7. (a) Allow the specimen to come to moisture equilibrium with the atmosphere of the balance case. Weigh, to the nearest milligram, 1.5 g. of the specimen for the alpha-cellulose determination. Weigh at the same time samples for the moisture and ash determinations, and also for those determinations of components other than cellulose as may be found necessary for calculation of the total cellulose content, such as fillers and sizing materials.

(b) Perform all operations and keep all liquids, as nearly as possible, at  $20 \pm 0.1$  C.

(c) Add 100 ml. of NaOH (17.5 per cent) to the sample in a 400-ml. beaker. Macerate until uniformly wet and dispersed, and let stand 10 min. from the time of addition of NaOH. Dilute with 165 ml. of water, stir the mixture thoroughly, and let stand 1 hr. longer, stirring once during the interval. After stirring once more, pour the mixture upon a medium-weight cotton cloth cut to fit a 7.5-cm. Büchner funnel, the cloth having been previously washed, and the

fitted piece dried to constant weight at 100 to 105 C. and weighed in a weighing bottle before use. Remove by refiltration any fibers passing through the filter. Before the last of the liquid has run through, add water, break up the pad well with a pointed stirring rod, and keep it in a loose condition until 200 ml. of wash water have passed through. Then cover the alpha-cellulose with acetic acid (10 per cent) and allow to soak for 5 min., after which pass an additional 500 ml. of wash water through while breaking up the pad with a pointed stirring rod.

(d) Dry the alpha-cellulose on the cloth in the original weighing bottle overnight at 100 to 105 C., cool, and weigh. Determine the ash content and correct the weight accordingly. The sizing materials, if any, remaining with the alpha-cellulose are taken as 0.25 per cent glue, 0.2 per cent starch, and 0.2 per cent resin, based upon the dry weight of the test specimen, and the corresponding weights subtracted from the total.

##### **Report**

8. The percentage of alpha-cellulose shall be based upon the total cellulose including pentosans, but excluding moisture, ash, resin, or any sizing or other added nonfibrous materials. All determinations of alpha-cellulose shall be made in duplicate, the results of which shall agree at least within 0.4 per cent of the dry weight of the sample, and the average shall be expressed to the nearest 1 per cent.

NOTE.—The precision of the volumetric method is much greater than these requirements would indicate. It is felt, however, that the average value of two determinations which differ by 0.4 when expressed as a percentage, adequately represents the specimen as prepared for testing. When, however, a given specimen is ground and tested in different laboratories, greater disagreement may arise, due to the

differences in freeness and extent of fibrillation of the fibers on the one hand, and individual differences in analysts on the other, added to the lack of perfect uniformity of the test specimen, and ordinary errors in analysis. Therefore, the reporting of alpha-cellulose values to the nearest 1 per cent is more compatible with experience, and is sufficient for the practical usefulness of the value. The same remarks apply to beta- and gamma-cellulose values.

#### BETA- AND GAMMA-CELLULOSE

##### Procedure

9. Use the remainder of the filtrate from the volumetric alpha-cellulose procedure (Section 5) for this determination. Acidify with 15 to 16 ml. of 6  $N$   $H_2SO_4$  and, after cooling, dilute the mixture to 100 ml., pour into a cylinder, and let stand at room temperature until the beta-cellulose has settled, which is usually overnight. Then remove 50 ml. of the supernatant liquid, oxidize, and titrate as before.

##### Calculations

10. (a) Calculate the percentage of gamma-cellulose by substitution in the

following Eq. 4, after applying corrections for any glue and starch, but not resin, in the same manner as described for the alpha-cellulose in Section 6 (b).

$$\text{Gamma-cellulose, per cent} = \frac{4C}{A + B} \times 100. (4)$$

where:

$C$  = milliliters of  $K_2Cr_2O_7$  required to oxidize the gamma-cellulose,

$A$  = milliliters of  $K_2Cr_2O_7$  required to oxidize the alpha-cellulose (Eq. 1), and

$B$  = milliliters of  $K_2Cr_2O_7$  required to oxidize the filtrate (Eq. 2).

(b) Calculate the percentage of beta-cellulose by subtracting the sum of the percentages of alpha-cellulose and gamma-cellulose from 100.

##### Report

11. Beta-cellulose and gamma-cellulose shall be reported on the same basis as prescribed for alpha-cellulose in Section 8.

*Standard Method for*  
QUANTITATIVE DETERMINATION OF COATING  
ON MINERAL-COATED PAPER<sup>1</sup>



A.S.T.M. Designation: D 687 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 687; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method covers the procedure for the quantitative determination of the coating on the ordinary types of mineral-coated paper, in which casein (or other proteins) or starch, is the adhesive. It is not intended for testing special types of coated paper, in which lacquers, varnishes, or similar materials are used.

### Reagent

2. *Enzyme Solution.*—Prepare an aqueous solution containing approximately 1.5 g. of a suitable enzyme (Notes 1 and 2) and 25 ml. of 0.1 *N* NaOH per liter.

NOTE 1.—The enzyme recommended by Sutermeister and Porter<sup>3</sup> is trypsin, but some of the mixtures of enzymes used commercially for desizing cotton and degumming silk have been found to be more rapid in action, less expensive, and more stable.<sup>4</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

<sup>3</sup> E. Sutermeister and L. W. Porter, "New Wrinkles in Paper Testing," *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Series XIII, Vol. 1, p. 206 (1930).

<sup>4</sup> The preparation "Degoma D. L.," made by Röhm & Haas Co., 222 W. Washington Square, Philadelphia 5, Pa., is particularly suitable.

NOTE 2.—While an enzyme may not be essential if starch is the only adhesive used, it will not interfere with the removal of the coating in such cases, and it is essential where casein is the binder.

### Test Specimens

3. The test specimen shall be cut from the sample in such a way to be thoroughly representative of the paper. The specimen shall consist of a sheet of the paper not less than 25 sq. in. in area.

### Test Conditions

4. All test specimens shall be brought to a standard condition, prior to testing, in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials.<sup>5</sup> Measurement and weighing of the specimens shall be done under the standard atmospheric conditions.

### Procedure

5. (a) Measure the area of the test specimen with an accuracy of plus or minus 0.5 per cent. (If the test speci-

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

men is 5 by 5 in., this requires measuring the length and width to the nearest 0.025 in. (about  $\frac{1}{8}$  in.). Weigh the test specimen with an accuracy of plus or minus 0.5 per cent. (For lightly coated papers, this requires weighing to the nearest 0.005 g.; for heavily coated papers, it may require weighing only to the nearest 0.05 g.) From these data, calculate the basis weight of the paper according to the Standard Method of Test for Basis Weight of Paper and Paper Products (A.S.T.M. Designation: D 646) of the American Society for Testing Materials.<sup>5</sup>

(b) Lay the specimen in a flat-bottomed tray, preferably of glass, with the coated side of the specimen up. Cover the specimen completely with the enzyme solution and allow to stand at least 1 hr. at 50 C. If the paper is coated on both sides, pour off the enzyme solution, reverse the specimen in the tray, re-cover with the enzyme solution, and again allow to stand for at least 1 hr. at 50 C. Remove the test specimen from the enzyme solution, lay it on a plane glass surface, and brush off the coating, using a camel's hair brush and taking care not to dislodge paper fibers. Additional treatment with the enzyme solution and heating may be required for very resistant coatings.

(c) After the coating appears to be entirely removed, stand the glass pane with the test specimen on it at a slight angle and, using a wash bottle, wash each side of the specimen with water

while it is held against the glass by one corner. When the specimen appears to be thoroughly washed, dry it at room conditions, then allow it to come to equilibrium with the standard atmosphere, and again weigh with an accuracy of plus or minus 0.5 per cent. The difference between the weights of the coated and decoated specimens is the amount of coating material.

### Report

6. The report shall include the following:

(1) The amount of coating expressed as a percentage by weight of the conditioned paper,

(2) The amount of coating expressed as pounds per standard ream of 500 sheets, 25 by 40 in. in size,<sup>6</sup> of the conditioned paper, and

(3) The weight of the de-coated, conditioned paper on the same weight basis as Item (2).

### Precision

7. Because of the variable nature of coating materials, the precision of the results obtained by this method may vary. With most ordinary types of coating, the percentage of coating material found should be correct to within 0.5 to 2.0.

<sup>5</sup> The results based on this standard ream can be readily converted to any trade custom ream desired by means of the calculations described in the Standard Method of Test for Basis Weight of Paper and Paper Products (A.S.T.M. Designation: D 646), which appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# Standard Method of CONDITIONING PAPER AND PAPER PRODUCTS FOR TESTING<sup>1</sup>



A.S.T.M. Designation: D 685 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 685; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method covers the procedure for conditioning specimens of paper and paper products prior to testing.

NOTE.—The procedure for conditioning specimens of container grades of paperboard, corrugated board and solid fiberboard, and containers made from such grades of paperboard is covered in the Standard Method of Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (A.S.T.M. Designation: D 641) of the American Society for Testing Materials.<sup>3</sup>

## Standard Condition

2. Standard condition shall be that obtained in a circulating atmosphere maintained at a relative humidity of  $50 \pm 2$  per cent and a temperature of  $23 \pm 2$  C. ( $73 \pm 3.5$  F.) (Note), except that the variation in temperature alone shall not be of such magnitude as to cause a variation in relative humidity greater than that specified.

NOTE.—For very precise work, closer limits on relative humidity and temperature may be necessary.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## Apparatus

3. The apparatus shall consist of the following:

(a) *Preconditioning Equipment*.—A desiccator or other suitable means for reducing the moisture content of the specimens before conditioning in accordance with Section 6.

(b) *Conditioning Room*.—A conditioning room or chamber that can be accurately controlled to a relative humidity of  $50 \pm 2$  per cent and a temperature of  $23 \pm 2$  C. ( $73 \pm 3.5$  F.).

(c) *Psychrometer*.—Either a sling psychrometer, or a stationary type of psychrometer having the air circulated over the thermometer bulbs mechanically.

NOTE.—It is preferable to draw the air over the thermometer bulbs rather than to blow it over them, as, in the latter case, the heat of the fan causes errors in the readings.

(d) *Thermometers*.—It is recommended that the thermometers conform approximately to the following requirements: Range, 0 to 50 C. (32 to 122 F.); graduation, 0.2 C. (0.5 F.). They should be matched to within 0.1 C. (0.2 F.) throughout the range used. Under ordinary conditions, an error of



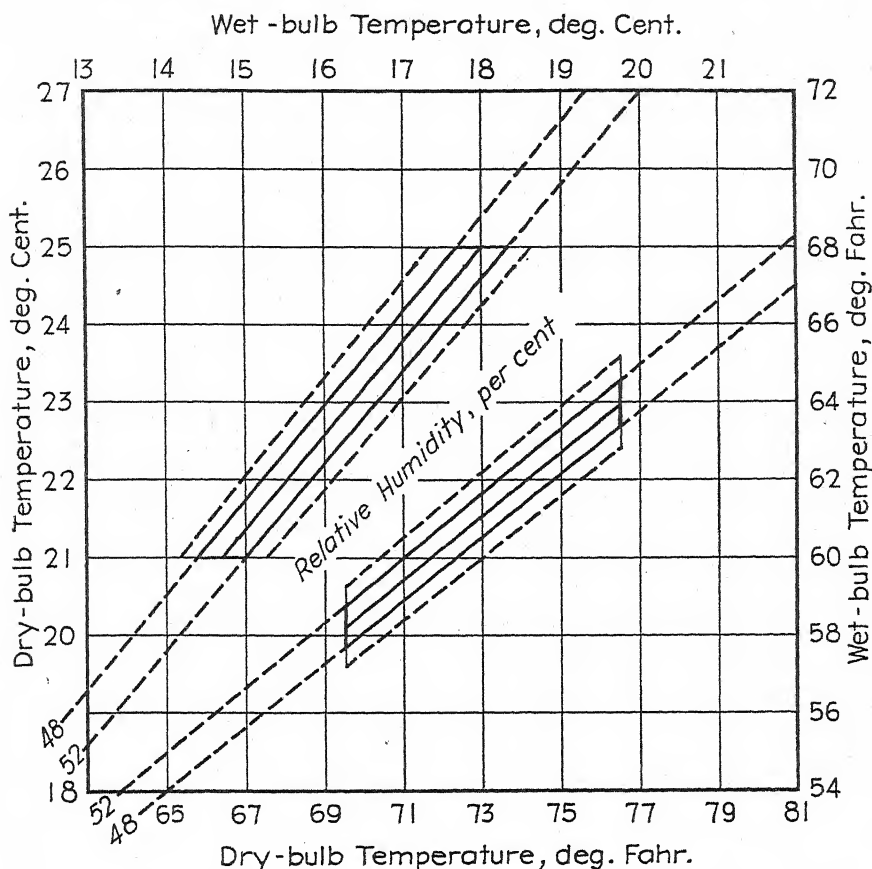


FIG. 1.—Psychrometric Chart for Determining Relative Humidity.

NOTE.—This psychrometric chart provides a convenient means for determining the relative humidity (in the neighborhood of 50 per cent relative humidity and 23 C. (73 F.) temperature) from readings of a ventilated wet- and dry-bulb psychrometer. Permissible temperature and relative humidity conditions lie within the solid-line parallelogram. The chart is based on a barometric pressure of 760 mm. (30 in.) of mercury. For each 900 ft. above sea level add 0.5 to the percentage relative humidity.

Example.—Dry-bulb temperature, 24.2 C.; wet-bulb temperature, 17.5 C.; elevation, 1700 ft. above sea level. The corresponding relative humidity is 51 (from chart) +  $\left(\frac{1700}{900} \times 0.5\right) = 52$  per cent.

1 per cent in the relative humidity determination corresponds to an error of approximately 0.1 C. (0.2 F.) in the wet-bulb depression.

#### Calibration of Thermometers

4. The thermometers used for determining humidity and temperature (Note) shall be accurately calibrated by comparison with certified standard thermometers, and any corrections found necessary applied to the readings.

#### Determination of Relative Humidity and Temperature

5. (a) Determine the relative humidity of the conditioning atmosphere by means of either a sling psychrometer or a stationary type of psychrometer. In both cases, the circulation of air around the thermometer bulbs should be at the rate of not less than 3 m. (10 ft.) per sec., and the exposure not less than 60 sec., before the readings are taken. When the sling type psychrometer is



used, make the readings, especially of the wet bulb, as quickly as possible after bringing it to rest.

(b) From the wet-bulb and dry-bulb readings, determine the relative humidity from the psychrometric chart shown in Fig. 1.

NOTE.—The wet-bulb temperature,  $T_w$ , in degrees Fahrenheit corresponding to a relative humidity of 50 per cent at a given air temperature,  $t$ , in degrees Fahrenheit (dry-bulb), and a given barometric pressure,  $B$ , in inches of mercury, may be calculated as follows:

$$T_w = 0.827t + 0.72 - 0.15(30 - B)$$

#### Procedure

6. (a) Suspend each test specimen so that the conditioning atmosphere will have free access to all its surfaces. Means shall be provided for so circulating the air of the conditioning and testing chamber that its humidity and temperature will be uniformly maintained. The conditioning time shall be sufficient for the moisture content of the specimen to attain equilibrium with

the conditioning atmosphere, as determined by conditioning to constant weight by accurate weighings at intervals of not less than 30 min. (Notes 1 and 2).

NOTE 1.—With good circulation, a conditioning period of 4 hr. is usually sufficient for papers of ordinary weight and composition, but some hard-sized papers, boards, and water-resistant specialties may require 24 hr. or longer.

NOTE 2.—After the test specimens are conditioned, they should be handled as little as possible and not breathed on.

(b) For work of such precision that the hysteresis in the equilibrium moisture content may lead to an appreciable error, the moisture content equilibrium under standard conditions shall be approached from a drier state by first reducing, if necessary, the moisture content to less than half the value under standard conditions, and then conditioning under standard conditions. For that purpose, the specimens may be dried in a desiccator or by other convenient means, provided the temperature does not exceed 60 C. (140 F.).

# Standard Method of CONDITIONING PAPERBOARD, FIBERBOARD, AND PAPER- BOARD CONTAINERS FOR TESTING<sup>1</sup>



A.S.T.M. Designation: D 641 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 641; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

## Scope

1. This method covers the procedure for conditioning specimens of container grades of paperboard, corrugated board and solid fiberboard, and containers made from such grades of paperboard, prior to testing.

## Standard Condition

2. Standard condition shall be that obtained in a circulating atmosphere maintained at a relative humidity of  $50 \pm 2$  per cent and a temperature of  $73 \pm 2$  F., except that the variation in temperature alone shall not be of such magnitude as to cause a variation in relative humidity greater than that specified.

## Apparatus

3. The apparatus shall consist of the following:

(a) *Drying Oven*.—A drying oven, electric or other suitable type, of suffi-

cient size and capacity to permit the preconditioning of the specimens to a moisture content below that which will be attained by the later conditioning.

(b) *Conditioning Room*.—A conditioning room or chamber which can be accurately controlled to a relative humidity of  $50 \pm 2$  per cent and a temperature of  $73 \pm 2$  F.

## Procedure

4. (a) Each specimen to be tested shall first have its moisture content initially reduced, if necessary, so that when it is exposed to the standard condition, its moisture content will approach its equilibrium condition by taking on moisture from the atmosphere. This shall be accomplished by exposing the specimen in a hot room or preconditioning room, the temperature and humidity of which are such as to cause the specimen to give off moisture. The duration of exposure in the preconditioning room shall be such that the moisture content of the specimen when removed from the room is below the moisture content which will

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1943.

result after exposure in the constant humidity room.

(b) Each specimen shall then be so exposed that the circulating air at the standard condition will have free access to all its exterior surfaces.

(c) The minimum time of exposure of the specimen in the standard conditioning atmosphere before it is tested shall be

5 hr. for specimens of paperboard, corrugated board, fiberboard, and unsealed boxes; the minimum time of exposure for sealed fiberboard boxes shall be 16 hr.

#### Report

5. The moisture content of the specimen at the time of test shall be reported.

## *Standard Method of Test for* FLAMMABILITY OF TREATED PAPER AND PAPERBOARD<sup>1</sup>



A.S.T.M. Designation: D 777 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 777; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test for flammability is applicable to papers and paperboards of all types not more than  $\frac{1}{16}$  in. in thickness, that have been treated to prevent the spread of the flame when ignited. Such papers are commonly designated as flameproofed.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Metal Frame*.—A metal frame designed to hold a specimen  $2\frac{3}{4}$  in. in width and  $8\frac{1}{4}$  in. in length by gripping  $\frac{3}{8}$  in. of each long edge in a clamp. The specimen with an area 2 by  $8\frac{1}{4}$  in. thus exposed is held with the long axis in a vertical position.

(b) *Test Flame*.—A test flame supplied by a bunsen or Tirril gas burner having a tube  $\frac{3}{8}$  in. in inside diameter.

(c) *Stop Watch*.

### Test Specimens

3. The test specimen shall consist of a piece of the sample  $2\frac{3}{4}$  in. in width and  $8\frac{1}{4}$  in. in length. Specimens shall be cut in both principal directions of the sheet.

### Procedure

4. The test specimen shall be clamped in the metal frame with the long axis in a vertical position. The testing flame shall be adjusted to a height of  $1\frac{1}{2}$  in. with the air supply closed, taking precaution to protect it from draft. The test flame shall be applied to the center of the lower edge of the specimen at a level that will make the distance from the top of the burner tube to the edge of the specimen  $\frac{3}{4}$  in. The test flame shall be applied for 12 sec. and then withdrawn. The duration of afterglow in seconds after removal of the flame shall be noted. The height that is charred to the extent that it will break away readily when crumpled between the thumb and forefinger shall be measured. Not less than three

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

specimens shall be tested in each principal direction of the sheet.

NOTE.—Papers showing an average char length of more than  $4\frac{1}{2}$  in. or a maximum of more than  $5\frac{1}{2}$  in. for any one specimen are generally not classed as flameproof; however, it is not the intent that this shall constitute a specification for flammability of paper or paperboard.

### Report

5. The report shall include the following:

(1) The average height of the charred or burned areas in inches, measured from the bottom of the test specimen and reported as "char length," and

(2) Duration of afterglow in seconds.

## Standard Methods of Test for FOLDING ENDURANCE OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 643 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 643; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These methods of test for measuring the folding endurance of paper cover two test procedures, as follows:

Method A. Schopper Folding Endurance.

Method B. M.I.T. (Massachusetts Institute of Technology) Folding Endurance.

(b) The Schopper apparatus is applicable for testing papers having a thickness of not over 0.01 in. The M.I.T. apparatus can be adjusted for testing papers of any thickness. There is no constant relation between the values obtained with the two types of apparatus.

### METHOD A. SCHOPPER FOLDING ENDURANCE<sup>3</sup>

#### Apparatus

2. The apparatus shall consist of the following:

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1941 to 1943.

<sup>3</sup> For further details of this method reference should be made to the following articles:

F. P. Veitch, C. F. Sammet, and E. O. Read, "Folding Endurance of Paper," *Paper* 20, No. 12, 13, May 30, 1917.

W. Herzberg, "Resistance to Folding," *Chemical Abstracts*, Vol. 14, No. 2262, July 20, 1920.

L. W. Snyder and F. T. Carson, "Calibration and Adjustment of Schopper Folding Tester," Nat. Bureau Standards, *Technologic Paper*, No. 357, October 15, 1927.

"The Schopper Bending Endurance Tester," *Instrumentation Report*, No. 23, Inst. Paper Chemistry, *Paper Trade Journal* 109, No. 5, p. 35, August 3, 1939.

(a) *Schopper Tester*.—Two horizontally opposed clamps, approximately 10 cm. apart, provided with spring tension which varies during the folding cycle as a slotted folding blade, sliding back and forth between creasing rollers, folds the paper. The clamps, while in motion, shall be freely suspended between the tension springs, except that they shall be supported from below by rollers. The folding blade shall be 0.50 mm. (0.02 in.) in thickness, and the edges of the vertical folding slot shall be cylindrical and shall extend somewhat above and below the normal position of the test specimen. The four creasing rollers, each approximately 6 mm. (0.24 in.) in diameter and 18 mm. (0.71 in.) in length, shall be arranged symmetrically about the mid-position of the folding slot, and shall preferably be provided with jewel bearings.

(b) *Motor*.—A means of imparting harmonic motion of constant period to the reciprocating blade. A power-driven apparatus is preferable.

(c) *Counter*.—A device for registering the number of double folds, which stops automatically when the specimen is severed.

#### Calibration of Apparatus

3. (a) Test the clamps by fastening a specimen in place in the manner de-

scribed in Section 7, alternately applying and releasing the tension a number of times. Then, with tension released, note whether the specimen remains smooth and straight as originally inserted. Buckling or waviness indicates a faulty clamp which will allow the specimen to slip.

(b) Inspect all rollers for worn surfaces and for bearing friction, and make the necessary corrections. Adjust the supporting rollers so that they do not bind against the clamps in any position. With leaf gages inspect the four creasing rollers for parallelism and clearances. Also, make sure that the two edges of the folding slot are parallel with each other and with the creasing rollers. Adjust the distance between the folding blade and the two creasing rollers on each side to 0.38 mm. (0.015 in.), and the width between rollers of the space occupied by the unbent specimen to approximately 0.5 mm. (0.02 in.). As a final test of alignment, fold a specimen somewhat short of failure and inspect it for uniformity of wear along the crease. If the specimen seems weaker at one end of the crease than at the other, faulty alignment of the rollers or the folding slot is indicated (if the clamps have been properly adjusted), which may result in low values for folding endurance.

(c) The roller friction may be measured by means of the bell-crank-lever weighing device, as follows: First, set a pair of bow dividers to show the displacement of each clamp when loaded directly with 1 kg. Then, shift the weighing device 90 deg. so as to load a clamp through a ribbon passed around one of the creasing rollers. Add weights in excess of 1 kg. until the direct 1-kg. displacement is reproduced. This excess weight is a measure of the roller friction in terms of the increased tension it will produce. Repeat the measurement for

the other three rollers. The excess weight required shall not be greater than 100 g.

(d) Adjust the tension spring attached to the clamps against a dead-weight load so that the tension on the specimen during a test is 790 g. when the clamps are farthest apart (and when the specimen is straight and free) and 1 kg.  $\pm$  50 g. when they are nearest together. Make adjustments preferably on the assembled instrument with the aid of a suitable weighing device, such as a balanced bell-crank lever with knife-edge fulcrums at the center of gravity, capable of balancing the tension of a horizontal spring against the weight of a known mass. Fasten a strip of strong paper or celluloid, about 0.005 in. (0.127 mm.) in thickness in the clamps and apply the tension. Set a pair of bow dividers (by spanning the distance between two suitably placed fiducial marks, such as small punch marks) to show the displacement of each clamp. With a load of 790 g. acting on one clamp and spring, adjust the spring until this displacement is reproduced. Repeat for the other spring. To verify the tension at maximum displacement, set a pair of dividers to show the displacement of each clamp when the folding blade has pushed the crease in the specimen to the end of its stroke each side of the midposition (four measurements). With the aid of the weighing device, load each clamp until this displacement is reproduced in each case. The load required in each case should be approximately 1 kg.

(e) Apparatus in steady use shall be adjusted and calibrated at intervals of not more than 1 month.

### Sampling

4. The material shall be sampled in accordance with the Standard Method

of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>4</sup>

#### Test Specimens

5. Test specimens  $15 \pm 0.25$  mm. ( $0.59 \pm 0.01$  in.) in width and 10 cm. (4 in.) in length shall be cut accurately from the sample in each principal direction of the paper. The specimens shall be initially free from folds, wrinkles, or blemishes not inherent in the paper, and the area in which the flexing is to take place shall not contain any portion of the watermark. The edges of the specimens shall be clean-cut and parallel to the opposite edge. At least 10 specimens cut from each principal direction of the paper shall be tested.

#### Test Conditions

6. All test specimens shall be brought to a standard condition, prior to testing, in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials,<sup>4</sup> and all tests shall be made under standard atmospheric conditions.

#### Procedure

7. With the vertical slot of the reciprocating blade in its central position, place the specimen in the slot and fasten the ends firmly and squarely in the clamps with the surface of the specimen lying wholly within one plane. Handle the specimen by the ends and do not touch it with the hands in the region which is to be folded. Then apply the prescribed tension and fold the specimen at a uniform rate of approximately 120 double folds per minute until it is severed at the crease. Record the number of double folds required to sever the specimen.

#### Report

8. The number of double folds required to sever the specimen shall be reported as Schopper folding endurance (double folds) and shall include the number of tests, and the average, maximum, and minimum number of folds for each of the principal directions of the paper. Specimens tested cut with their length in the machine direction of the paper shall be designated as "machine direction." Specimens tested with their length cut at right angles to the machine direction of the paper shall be designated as "cross machine direction." In reporting average results all digits after the first two shall be rounded off to zero.

#### METHOD B. M.I.T. FOLDING ENDURANCE<sup>5</sup>

#### Apparatus

9. The apparatus shall consist of the following:

(a) *M.I.T. Tester*.—A loading clamp constrained to move without rotation in a direction perpendicular to the axis of rotation of the folding head and having its clamping surfaces in the plane of this axis. The load shall be applied through a spring attached to the loading clamp which shall be easily adjustable to provide any desired tension on the specimen from 0 to 1.5 kg. The deflection of the spring when loaded shall not be less than 17 mm. (0.67 in.) per kg.

An oscillating folding head which shall support two smooth, cylindrical folding surfaces parallel to, and symmetrically placed with respect to, the axis of rotation. The position of the axis of rotation shall be approximately in the common tangent plane to the two folding surfaces in the conventional design, and midway between them. The folding

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>5</sup> For further details of this method reference should be made to a paper by L. W. Snyder and F. T. Carson, "A Study of the M.I.T. Paper Folding Tester," *Paper Trade Journal* 96, No. 22, p. 40, June 1, 1933.



head shall be provided with a clamping device back of the axis of rotation and so designed that no clamping pressure is exerted nearer than  $\frac{3}{8}$  in. (3.18 mm.) to the bending axis. The rotary oscillating movement of the folding clamp shall be such as to fold the paper through an angle of  $135 \pm 5$  deg. to both right and left of the position of zero fold. Each of the two folding surfaces shall have radius of curvature of  $0.38 \pm 0.015$  mm. ( $0.015 \pm 0.001$  in.) and a length of not less than 19 mm. (0.75 in.). The distance separating the folding surfaces shall be greater than the uncompressed thickness of the paper being tested, but shall not exceed it by more than 0.25 mm. (0.01 in.).

(b) *Motor*.—A motor-driven device for imparting a rotary oscillating motion of constant period to the folding clamp.

(c) *Counter*.—A device for registering the number of double folds required to sever the specimen.

#### Calibration

10. (a) All working parts of the apparatus shall be in good condition, well oiled, and in proper adjustment. Particular care shall be given to make certain that the folding edges are free from rust or dirt.

(b) Measure the plunger friction by determining the additional load required to move the plunger perceptibly when displaced under a load of 1.0 kg. or the load tension used in testing. The additional load required shall not be greater than 25 g.

(c) Measure the change in tension due to eccentricity of rotation of folding edges as follows: Place a strip of strong paper, cut in the machine direction and of the proper thickness, in the tester in the same manner in which a folding test would be made, and apply a tension of 1.0 kg. or that prescribed for the test. Rotate the folding head slowly through-

out the entire folding cycle and measure the maximum change in displacement of the plunger to an accuracy of 0.1 mm. (0.004 in.). The amount of load required to produce the same displacement shall not be greater than 35 g. Measure the curvature of the folding edges by comparing suitable casts magnified in profile to standard circles.

(d) Apparatus in steady use shall be adjusted and calibrated at intervals of not more than 1 month.

#### Sampling

11. The material shall be sampled in accordance with the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>4</sup>

#### Test Specimens

12. Test specimens  $15 \pm 0.25$  mm. ( $0.59 \pm 0.01$  in.) in width and at least 14 cm. (5.5 in.) in length shall be cut accurately from the sample in each principal direction of the paper. The specimens shall be initially free from folds, wrinkles, or blemishes, and the area in which the flexing is to take place shall not contain any portion of the watermark. The edges of the specimens shall be clean-cut and parallel to the opposite edge. At least 10 specimens cut from each principal direction of the paper shall be tested.

#### Test Conditions

13. All test specimens shall be brought to a standard condition prior to testing in accordance with A.S.T.M. Method D 685,<sup>4</sup> and all tests shall be made under standard atmospheric conditions.

#### Procedure

14. Place the oscillating folding head in the position of zero fold. Place on top of the plunger a weight equivalent to the tension desired on the specimen and clamp the plunger in position when depressed under this load. Then clamp

the specimen firmly and squarely in the jaws with the surface of the specimen lying wholly within one plane and not touching the jaw mounting-plate. Handle the specimen by the ends and do not touch it with the hands in the region which is to be folded. Then apply the prescribed tension to the specimen by releasing the plunger. If the reading of the load indicator has changed, reset it by means of the adjusting screw to agree with the reading obtained when the weight was applied. Whenever possible a tension of 1.0 kg. shall be used, but if this does not afford practical test results, a greater or a lesser tension may be used. Fold the strip at a uniform rate of 175 double folds 15 per cent per minute until it is severed at the crease.

Record the number of double folds required to sever the specimen.

#### Report

15. The number of double folds required to sever the specimen shall be reported as the M.I.T. Folding Endurance (double folds) *at the tension used*, and shall include the number of tests, and the average, maximum, and minimum number of folds for each for the principal directions of the paper. Specimens tested with their length cut in the machine direction shall be designated as "machine direction." Specimens tested with their length cut at right angles to the machine direction shall be designated as "cross machine direction." In reporting average results all digits after the first two shall be rounded off to zero.

## *Standard Method of Test for*

### EFFECT OF HEATING ON FOLDING ENDURANCE OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 776 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 776; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. (a) This method of test covers the procedure for determining the effect of heating on the folding endurance of paper.<sup>3</sup> This test may be used for most papers, but it is not applicable to papers containing large amounts of ground-wood fibers.

(b) This test determines how well paper retains its folding endurance after it has been heated under the conditions specified herein. The heating conditions specified in this method accelerate some of the deteriorative chemical reactions that cause brittleness of the paper.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

<sup>3</sup> For further information on this method the following references should be consulted:

R. H. Rasch, "Accelerated Aging Test for Paper," *Journal of Research*, Nat. Bureau Standards, Vol. 7, p. 466 (1931) (*Research Paper 352*).

B. W. Scribner, "A Comparison of Accelerated Aging of Record Papers with Normal Aging for Eight Years," *Journal of Research*, Nat. Bureau Standards, Vol. 23, p. 405 (1939) (*Research Paper 1241*).

A. E. Kimerly and B. W. Scribner, "Summary Report on the Bureau of Standards Research on the Preservation of Records," *Miscellaneous Publication M154*, Nat. Bureau Standards (1937).

P. F. Wehmer, "Effect of Volume of Fresh Air in Heat Test for Stability of Paper," *Paper Trade Journal*, Vol. 94, No. 3, January 28, 1932, p. 37.

R. H. Rasch and G. O. Stone, "Estimating Stability of Paper by Heating," *Paper Trade Journal*, Vol. 95, No. 4, July 28, 1932, p. 28.

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Oven*.—An oven of the mechanical circulation air type and of such design that it will maintain a temperature throughout the interior of  $105 \pm 2$  C. A continuous temperature recording device is necessary.

(b) *Folding Endurance Tester*.—A folding endurance tester of either the Schopper type or the M.I.T. type, as specified in the Standard Methods of Test for Folding Endurance of Paper (A.S.T.M. Designation: D 643).<sup>4</sup>

#### Test Specimens

3. The test specimens shall consist of strips 15 mm. (0.59 in.) in width, and of sufficient length to fit in the jaws of the folding tester. Not less than 20 strips each shall be used for the control test and for the heat treatment. They shall be cut in each principal direction of the paper and from not less than five different sheets of the

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sample of paper, and the control specimens and the specimens to be heated shall be cut alternately and contiguous to each other.

#### Procedure

4. (a) The test specimens to be heated shall be suspended in the oven in such a way that the air of the oven has free access to all surfaces of each strip, and that the strips shall not be in contact with any part of the oven. The specimens shall be heated at  $105 \pm 2$  C. for 72 hr. The oven shall be kept closed and no other materials shall be in the oven during the heating period.

(b) The heated and unheated test specimens shall be conditioned side by side in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685),<sup>4</sup> and shall be tested for folding endurance in the atmospheric conditions specified in Method D 685. The heated and un-

heated specimens shall be tested alternately in the folding machine in accordance with the procedure prescribed in A.S.T.M. Method D 643.

#### Report

5. The report shall include the following:

(1) The percentage of the original folding endurance retained after heating which shall be reported as "retention of folding endurance," and expressed as a percentage.

(2) Retention values (Note) reported for the machine direction, cross direction, and the average of both directions.

(3) The type of testing machine used, whether the Schopper or M.I.T. tester, and the tension applied in the case of the latter machine.

NOTE.—There is no constant relation between the test values obtained with the two types of testers, and retention values are not comparable unless all values were obtained with the same type of folding endurance testing machine.

## Standard Method of TURPENTINE TEST FOR GREASE RESISTANCE OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 722 - 45

ADOPTED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 722; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method covers the procedure for determining the grease resistance of paper by the turpentine test. It gives an accelerated comparison of the relative rates at which ordinary oils or greases, such as commonly found in foodstuffs, may be expected to penetrate papers, such as uncoated or unimpregnated greaseproof, glassine, and vegetable parchment.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Tube*.—A tube of any rigid material, 1 in. in inside diameter, not less than 1 in. in length, and with smooth ends.

(b) *Pipette*.—A pipette or medicine dropper, calibrated to deliver 1.1 ml.

(c) *Sand*.—A round-grained, natural silica sand<sup>3</sup> graded to pass a No. 20

(840-micron) sieve and be retained on a No. 30 (590-micron) sieve.

(d) *Book Paper*.—Sheets of coated, 80-lb., book paper,<sup>4</sup> 25 by 38 in., 500 basis, of convenient size.

### Reagent

3. *Turpentine (colored, water-free)*.—To 100 ml. of pure gum spirits of turpentine (c.p. grade, sp. gr. 0.860 to 0.875 at 60 F.), add 5 g. of anhydrous calcium chloride and 1.0 g. of oil-soluble red dye.<sup>5</sup> Stopper the container, shake well and let stand for at least 10 hr., shaking occasionally. Then filter through a dry filter paper at a temperature of approximately 70 F., and store in an airtight bottle.

### Sampling

4. The paper to be tested shall be sampled in accordance with the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>6</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1943 to 1945.

<sup>3</sup> Natural silica sand from the Ottawa Silica Co., Ottawa, Ill., is recommended.

<sup>4</sup> Cumberland Gloss No. 2, manufactured by S. D. Warren Co., Cumberland Mills, Maine, is recommended.

<sup>5</sup> E. I. du Pont de Nemours and Co. oil-soluble red dye is recommended.

<sup>6</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Test Specimens

5. (a) Not less than 30 representative test specimens 4-in. square shall be cut from the sample.

(b) The test specimens shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials.<sup>6</sup>

### Procedure

6. (a) Test not less than 30 conditioned specimens in the standard atmosphere prescribed in Tentative Method D 685, making an equal number of tests on each side of the sample. If possible, determine which is the felt side and which is the wire side of the specimen in accordance with the Standard Methods of Test for Wire and Felt Sides of Paper (A.S.T.M. Designation: D 725) of the American Society for Testing Materials.<sup>6</sup>

(b) Place each specimen on a sheet of the book paper which rests on a smooth, plane surface. Place the end of the tube on the specimen, and put 5 g. of sand in the tube. Since the purpose of the tube is solely to assure a uniform area of the sand pile, remove it immediately after the addition of the sand. Using the pipette or medicine dropper, add 1.1 ml. of the colored turpentine to the sand, and note the time.

(c) Move the test specimens to different positions on the book paper and examine the uncovered area of it for staining, every 30 sec for the first 2 min.,

every 1 min. for the next 8 min., and every 3 min. thereafter. As soon as the first red stain appears on the book paper, note the time. Record the elapsed time, in seconds, between the application of the turpentine and the appearance of the first definitely red stain as the transudation time.

NOTE.—In the absence of knowledge of the probable time of transudation, it is advisable to make a few preliminary tests.

### Report

7. (a) The report shall include the following:

(1) The number of specimens tested,  
(2) The maximum, minimum, and average turpentine transudation in seconds. If possible the results shall be reported separately for specimens tested with the felt side up and with the wire side up. All tests over 1800 sec. shall be reported as 1800+ sec. and if individual results of 1800+ sec. are included in any average, such average shall be followed by a plus sign. The average shall be reported on the basis of all tests made.

(b) It is recommended that the following report form be used in reporting results, when possible:

Turpentine Transudation, sec.	Felt Side Up	Wire Side Up
Maximum.....	1800+	1750
Minimum.....	1500	1400
Average of 15 tests.....	1750+	1600
Total average.....	1675+	

*Standard Method of Test for*  
HYDROGEN ION CONCENTRATION (pH) OF PAPER  
EXTRACT<sup>1</sup>



A.S.T.M. Designation: D 778 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 778; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Scope**

1. (a) This method of test covers the procedure for determining the hydrogen ion concentration of paper extract. In this method, the pH value of a paper corresponds to the hydrogen ion concentration of the filtered aqueous extract made of the cut or ground paper. Thus, the values obtained refer to the paper-water mixture and are designated as pH value and "H-ion" or "OH-ion" value of the paper.

(b) This method is suitable for the regular run of commercial and industrial papers, the water extracts of which are normally acidic and usually buffered. It is not satisfactory for determining the pH of unbuffered and neutral papers (Note 1), such as insulating papers. Insulating papers (Note 2) require a refined method which eliminates errors due to the absorption of carbon dioxide by the water extract during its preparation and testing.

NOTE 1.—Unbuffered papers are susceptible to changes in the hydrogen ion concentration (pH value) upon the addition of slight amounts of acids or bases to their water extracts. Neutral papers are neither acid nor alkaline in reaction. Their pH value is 7.0.

NOTE 2.—A method of test for pH of insulating paper appears in the Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (A.S.T.M. Designation: D 202).<sup>3</sup>

**Apparatus**

2. The apparatus shall consist of the following:

(a) *Disintegrator*.—A disintegrator, when required for use as in the case of thick paper, shall be of the Koerner type or its equivalent, and shall not contaminate or heat the paper (Note).

(b) *Glassware*.—Glassware which is resistant to acids and alkalis,<sup>4</sup> and shall include the following pieces:

(1) *Soil Digestion Flask*.—A soil digestion flask of 250-ml. capacity.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> Pyrex glass or its equivalent is recommended for this purpose.

(2) *Air Condensing Tube*.—An air condensing tube measuring 10 mm. in diameter and 100 cm. in length and equipped with a one-piece ground-glass stopper.

(c) *Electrometric pH Apparatus*.<sup>5</sup>—The electrometric pH apparatus shall consist of a glass electrode and a saturated potassium chloride-calomel half-cell capable of precision to within 0.1 pH, a potentiometer, and a galvanometer for measuring voltage. The apparatus shall be standardized frequently against 0.05 *M* potassium acid phthalate solution which has a pH of 4.0 over the entire range of room temperature.

NOTE.—If a hot plate is employed in the extraction, the suspension should not be boiled too vigorously or the water will be distilled off, thus concentrating the solution. If desired, a layer of asbestos paper may be placed on the hot plate to avoid overheating.

## Reagents

3. *Potassium Acid Phthalate Solution* (0.05 *M*).—Dissolve 10.2 g. of potassium acid phthalate sufficiently pure for acidimetric purposes in water and dilute to 1 liter. This solution generally will remain usable for several months at a pH of 4.0.

(b) *Distilled Water*.—Water containing ordinary amounts of CO<sub>2</sub>, but not more than corresponds to a pH of 5.9 may be used to test all papers including those in the neutral range between a pH of 6.0 to 7.0. A sample of the water shall be tested for alkaline impurities, by boiling for a few minutes, cooling, and then measuring its pH. If this is found to be higher than 7.3 the water shall be distilled from a solution containing approximately 1 g. of KMnO<sub>4</sub> and 4 g. of NaOH per liter. A double still-head is usually necessary.

<sup>5</sup> The complete electrometric pH apparatus is available commercially.

## Test Specimens

4. The test specimen shall be cut from the sample in such a way as to be thoroughly representative of it. Ordinary papers may be tested in the form of cuttings approximately 1 sq. cm. in area. If the paper is more than 0.012 in. in thickness, or very dense, it shall be reduced to cotton-like form in the disintegrator (Note). The prepared material shall be thoroughly mixed.

NOTE.—In case of doubt, a disintegrator shall be used. The use of a disintegrator should be avoided whenever possible because even with the greatest care, test specimens sometimes become contaminated in the disintegrator sufficiently to cause erroneous results.

## Procedure

5. (a) Weigh 1.0 g. of air-dry, cut, or ground test specimen, and transfer to a 250-ml. soil-digestion flask. Add a portion of 250 ml. of boiling distilled water and macerate with a flattened stirring rod until the specimen is uniformly wet. Then add the remaining portion of the distilled water, stir well, and affix to the flask a one-piece, ground-glass stopper and air-condensing tube, approximately 10 mm. in diameter and 100 cm. in length. Place the flask, connected with the air-condenser tube, in a heating bath or upon a hot plate, and maintain the contents of the flask at 98 to 100 C. Heat at this temperature for 1 hr., while shaking occasionally. At the end of the extraction period, remove the air-condensing tube, shake the flask, and filter the contents through a fritted-glass crucible with strong suction. Disconnect the suction just before the filtration is completed. Pour the filtrate into a clean flask, close it tightly with a clean rubber stopper, set aside in a cold-water bath, and cool to room temperature.

(b) Measure the pH of the cooled filtered mixture with the glass electrode.



Carefully wash the glass electrode, first with distilled water and then with the solution to be tested prior to the electrometric measurements. It may be necessary to rinse the electrode as many as 10 times with fresh portions of the extraction solution before successful readings of the same are obtained. Take readings until the voltage is constant to about 1 mv. for a period of several minutes. If the glass electrode had just previously been used for measurements in the alkaline region, and if the paper to be tested is in the neutral region, immerse the electrode in 0.05 *M* potassium acid phthalate for 1 min. and rinse before use. Make all tests in duplicate.

#### Calculation

6. Calculate the pH value of the paper extracts from the voltage readings by the following equations (Note):

$$\text{pH}_x = \text{pH}_b + \frac{E_x - E_b}{K}$$

or

$$\text{pH} = 4.0 + \frac{E_x - E_b}{0.05914}$$

where:

$\text{pH}_x$  = hydrogen ion concentration of the unknown solution,

$\text{pH}_b$  = hydrogen ion concentration of the buffer solution (potassium acid phthalate) used for calibration,

$E_x$  = voltage reading for the unknown solution,

$E_b$  = voltage reading for the buffer solution (potassium acid phthalate) used for calibration, and

$K = 0.00019832 \times (273.1 + \text{temperature in degrees Centigrade}).$

NOTE.—These equations shall be used for

measurements made at 25 C. Corrections should be made for measurements taken at other temperatures. The pH value may be conveniently read directly from graphs constructed upon the basis of these equations. A number of glass electrode instruments give readings directly in pH values.

#### Report

7. The average of the results shall be reported and expressed to the nearest

TABLE I.—CORRESPONDING pH, H-ION, AND OH-ION VALUES.

pH	H-ion Concentration	pH	OH-ion Concentration
4.0.....	$1000 \times 10^{-7}$	7.0.....	$1 \times 10^{-7}$
4.1.....	790	7.1.....	1.3
4.2.....	630	7.2.....	1.6
4.3.....	500	7.3.....	2
4.4.....	400	7.4.....	2.5
4.5.....	320	7.5.....	3
4.6.....	250	7.6.....	4
4.7.....	200	7.7.....	5
4.8.....	160	7.8.....	6
4.9.....	130	7.9.....	8
5.0.....	100	8.0.....	10
5.1.....	79	8.1.....	13
5.2.....	63	8.2.....	16
5.3.....	50	8.3.....	20
5.4.....	40	8.4.....	25
5.5.....	32	8.5.....	32
5.6.....	25	8.6.....	40
5.7.....	20	8.7.....	50
5.8.....	16	8.8.....	63
5.9.....	13	8.9.....	79
6.0.....	10	9.0.....	100
6.1.....	8	9.1.....	130
6.2.....	6	9.2.....	160
6.3.....	5	9.3.....	200
6.4.....	4	9.4.....	250
6.5.....	3	9.5.....	320
6.6.....	2.5	9.6.....	400
6.7.....	2	9.7.....	500
6.8.....	1.6	9.8.....	630
6.9.....	1.3	9.9.....	790
7.0.....	1	10.0.....	1000

0.1 pH, together with their corresponding arithmetical equivalents to denote "Active Acidity" H-ion or "Active Alkalinity" OH-ion. The corresponding values are given in Table I, rounded off to significant figures.

#### Reproducibility

8. Duplicate determinations should agree with 0.1 pH.

#### EXPLANATORY NOTE

NOTE.—For further information on this method the following references should be consulted:

J. O. Burton and R. H. Rasch, "Determination of the Alpha-Cellulose Content and Copper

Number of Paper," *Journal of Research*, Nat. Bur. Standards, Vol. 6, No. 4, p. 603 (1931).

B. L. Browning and R. W. K. Ulm, "The Nature and Measurement of Paper Acidity," *Paper Trade Journal*, Vol. 102, No. 8, February 20, 1936, pp. 69-89.

H. F. Launer, "Determination of the pH Values of Paper," *Journal of Research*, Nat. Bur. Standards, Vol. 22, p. 553 (1939) (*Research Paper RP 1205*).

Everett L. Wallace, "Method for Measuring the pH of Leather Using a Simple Glass Electrode Assembly," *Journal*, Am. Leather Chemists Assn., Vol. 30, No. 7, July, 1935, pp. 370-379.

L. M. Booth, "A More Rational Method of Stating Hydrogen Ion Concentration," *Paper Trade Journal*, Vol. 108, No. 8, February 23, 1939, p. 116; *Technical Association Papers*,

Tech. Assn. Pulp and Paper Industry, Series XXII, No. 1, June, 1939, p. 189.

Lewis B. Miller, "The Expression of pH as An Arithmetic Quantity," *Paper Trade Journal*, Vol. 109, No. 12, September 21, 1939, p. 22.

H. F. Launer, "Retention of Aluminum Ion and Hydrogen Ion in Papers," *Journal of Research*, Nat. Bur. Standards, Vol. 23, p. 663 (1939) (*Research Paper RP 1262*).

B. L. Wehmhoff, "The Determination of pH Values and Total Acidity of Paper," *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Series XIII, May, 1930, p. 231; Series XIV, May, 1931, p. 387.

P. F. Wehmer, "Hydrogen Ion Concentrations and Pentosans," *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Series XXIII, June, 1940, pp. 56 and 446.

## *Standard Method of Test for* Kerosine Number of Roofing and Flooring Felt BY THE VACUUM METHOD<sup>1</sup>



A.S.T.M. Designation: D 727 - 45

ADOPTED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 727; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test for the absorptive qualities of felt, expressed as kerosine number, is applicable to felts used in the manufacture of roofing and flooring.

NOTE.—The kerosine number of a felt, as determined by this method, is based on the relation between the specific gravity of the kerosine used and the specific gravity of water (1.00). To obtain the relation between the saturating capacity of the felt and any bituminous saturant to be used with the felt, the specific gravity at 77 F. (25 C.) of the bitumen shall be determined in accordance with the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cements, and Soft Tar Pitches (A.S.T.M. Designation: D 70) of the American Society for Testing Materials,<sup>3</sup> and that figure multiplied by the "kerosine number" of the felt, and expressed as a percentage figure.

### Kerosine Number

2. The kerosine number of roofing and flooring felt is calculated from the

maximum weight of a kerosine oil, of known specific gravity, retained by the felt after displacement of all the air from the interior voids. It is a measure of the amount of saturant which a given felt will absorb.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Balance*.—Analytical balance, sensitive to 1 mg.

(b) *Oven*.—Laboratory oven, steam or electrically heated and capable of maintaining a temperature between 220 and 225 F., inside dimensions to be not less than 12 by 12 by 12 in.

(c) *Weighing Container*.—Lightweight glass or metal container with tight-fitting cover for weighing the specimens, and of a suitable size to hold the specimens in a horizontal position without bending or distorting them.

(d) *Thermo-Hydrometer*.—Glass hydrometer and thermometer combined, also a glass hydrometer cylinder as prescribed in the Standard Method of Test for Gravity of Petroleum and

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1943 to 1945.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.<sup>3</sup>

(e) *Vacuum Pump*.—A vacuum pump capable of reaching and maintaining a vacuum of not less than 28 in. of mercury in the test cylinder.

(f) *Vacuum Cylinder*.—A glass vessel of approximately 1500 cu. cm. capacity for soaking the felt specimens in kerosine under vacuum.

(g) *Desiccator*.—A desiccator of size suitable for cooling felt strips in the weighing containers.

### Kerosine

4. The water-white kerosine used in the test shall have a specific gravity between 0.776 and 0.825 at 25 C. (77 F.).

### Test Specimens

5. Six representative specimens each measuring  $2 \pm \frac{1}{8}$  in. by  $5 \pm \frac{1}{8}$  in. in size shall be selected from the felt to be tested. The specimens shall be cut with the 5-in. side running parallel to the machine direction of the sheet.

### Procedure

6. (a) Place the test specimens in the tared weighing container and expose them, uncovered, for not less than 1 nor more than 2 hr. in the oven, the interior of which is maintained uniformly at a temperature between 220 and 225 F. The specimens shall be kept flat; they shall not be folded, rolled, or in any way distorted. Handle the six specimens as a unit in all of the following operations.

(b) Remove the specimens from the oven in the weighing container and place, still uncovered, in the desiccator to cool.

(c) Rapidly seal the weighing container with its cover and weigh the container and included specimens on the

analytical balance to the nearest 10 mg. Calculate the net weight of the dry specimens.

(d) Insert a wire hook in one corner of the specimens and immerse them at once in a vertical position in 1 liter of kerosine at  $25 \pm 1$  C. ( $77 \pm 1.8$  F.) in the glass vacuum vessel. Apply a vacuum of not less than 28 in. of mercury to the vessel containing the specimens and hold them under that condition for 15 min., or until the bubbles cease to come from the specimens, whichever period is the longer. Remove the specimens from the kerosine and permit them to drain in the machine direction of the felt fibers for 3 min.  $\pm 1$  sec., allowing the lower corner of each specimen to touch the edge of the kerosine container.

(e) Return the specimens to their weighing container, seal with its cover, and determine and record the combined weight of the specimens and the kerosine which they have absorbed.

(f) Determine the specific gravity at 25 C. (77 F.) of the kerosine used in the test in accordance with the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287) of the American Society for Testing Materials.<sup>3</sup>

### Calculation and Report

7. (a) Calculate the kerosene number of the specimens as follows:

$$\text{Kerosene number} = \left( \frac{b}{a} - 1 \right) \times \left( \frac{1}{g} \times 100 \right)$$

where:

$a$  = weight of dry felt,

$b$  = weight of felt plus absorbed kerosine, and

$g$  = specific gravity of kerosine at 25 C. (77 F.)

(b) *Report*.—Report the results of the kerosene number determinations to three significant figures.

## *Standard Method of Test for* MACHINE DIRECTION OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 528 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 528; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method describes test procedures for determining the machine direction of paper.

### Definitions

2. The two major directions of paper are defined as follows:

(a) *Machine Direction*.—The direction of the paper parallel to its forward movement on the paper machine.

(b) *Cross Direction*.—The direction of the paper at right angles to the machine direction.

### Test Specimens

3. (a) *Method 1*.—Circular test specimens shall be cut about 2 in. in diameter. For purposes of identification a straight line shall first be drawn through the section and adjacent parts of the paper from which the specimen is to be cut.

(b) *Method 2*.—Two test specimens shall be cut at right angles to each other, each 6 in. in length by  $\frac{1}{4}$  in. in width.

(c) *Method 3*.—No specially prepared test specimen is required for this test.

(d) *Method 4*.—Two test specimens shall be cut at right angles to each other, each 10 in. in length and of a proper width to fit a tension testing machine.

### Procedure

4. (a) *Method 1*.—The test specimen shall be floated on water and the direction of curl noted. The axis of curl is parallel to the machine direction of the paper.

(b) *Method 2*.—The two test specimens shall be held by the ends in a horizontal position, one over the other, placing first one and then the other on top. The specimen cut in the cross direction will bend more, and fall away from the specimen cut in the machine direction of the paper.

(c) *Method 3*.—The test specimen shall be burst in a manner similar to that employed when testing for bursting strength. The chief line of rupture will be at right angles to the machine direction of the paper.

(d) *Method 4*.—The two test specimens shall be broken in a manner similar

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

to that employed when testing for tensile strength. The specimen whose length corresponds to the machine direction of the paper will have the greater tensile strength.

NOTE.—If it is suspected that the length of neither specimen corresponds to the machine direction of the paper, other specimens should be cut at different angles, until one having the maximum strength is found.

*Standard Method of Test for*  
MOISTURE IN PAPER, PAPERBOARD, AND PAPERBOARD  
AND FIBERBOARD CONTAINERS<sup>1</sup>



A.S.T.M. Designation: D 644 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 644; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Scope**

1. This method covers the procedure for determining moisture in all papers, paperboards, and paperboard and fiberboard containers, except those containing matter other than water volatile at 100 to 105 C.

**Apparatus**

2. The apparatus shall consist of the following:

(a) *Weighing Container*.—Either a wide-mouth, glass-stoppered bottle or an airtight metal container, or any other suitable airtight container, depending on the size of the specimen required.

(b) *Thermometer*.

(c) *Drying Oven*.—A constant-temperature oven, equipped with means for insuring adequate temperature control and air circulation, and preferably equipped with means for drying the air entering the oven.

(d) *Chemical Balance*.—A chemical balance sensitive to 1 mg. for weighing

specimens of 2 g. and under, and sensitive to 0.05 per cent of the original weight of the specimen for larger specimens.

(e) *Desiccator*.

**Test Specimens**

3. (a) When the amount of moisture is determined for the purpose of calculating the results of a chemical analysis of paper on a moisture-free basis, the test specimens shall weigh not less than 1 g., and preferably not less than 2 g. each, and when weighed shall be in moisture equilibrium with the specimens being analyzed. When moisture is determined for the purpose of calculating the amount of moisture in a shipment of paper, the test specimens shall weigh not less than 50 g. each.

(b) When the moisture is determined for the purpose of calculating the amount of moisture in a shipment, the paper shall be sampled (Note) in accordance with the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1941 to 1944, being revised in 1942.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE.—In sampling a shipment for moisture content, extreme care must be taken to avoid change in moisture content during the sampling.

(c) When the moisture is determined on combined board or containers that are to be tested for other physical properties, test specimens shall weigh approximately 50 g. and shall be taken so as to be representative of the material being tested. Specimens shall be cut from unsealed sections and preferably from unprinted sections of the container, and shall be taken from each type of container being tested.

#### Procedure

4. (a) Handle large specimens with clean, dry rubber gloves. Transfer the specimens to the weighed container as soon as they are withdrawn from the package, and close the container immediately. If a delay of over a second or two in transferring the specimen to the container is unavoidable, keep the specimen covered on both sides with several adjacent layers of the paper from which it was withdrawn, until ready to place it in the container. Unless the specimen is later to be spread out in the oven, avoid filling the container tightly. After the specimen has been placed in a weighed container, weigh it in the closed container to obtain the wet weight of the test specimen (Note).

NOTE.—The weighing container or specimens should not be touched with the fingers during this test.

(b) Remove large specimens, unless the container has a removable basket, from the container in which they were weighed, spread them in a basket or tray which will permit free circulation of air around them, and place them as well as the original container in the oven. Heat for about 2 hr. at 100 to 105 C., replace the specimens in the original container,

and close it, doing this, if possible, without removing the specimens from the oven. Let the closed container and contents cool at room temperature, and weigh.

(c) Place small specimens in the drying oven, without removing them from the weighing bottle, remove the stopper of the bottle, heat for 1 hr. at 100 to 105 C., close the bottle in the oven, cool to room temperature in a desiccator, and weigh. Remove the stopper momentarily just before weighing to adjust any change in air pressure.

(d) Repeat the periodic drying and weighing of the specimen until the difference in weight between two successive weighings is not more than 0.1 per cent of the weight of the specimen.

(e) Make all weighings with the cover on the container and weigh to within 0.05 per cent of the total weight of the original specimens.

#### Calculation and Report

5. (a) When the percentage of moisture based on the original weight is required, it shall be calculated as follows:

$$\text{Moisture, per cent} = \frac{W_1 - W_2}{W_1} \times 100$$

where:

$W_1$  = original weight of the specimen,  
and

$W_2$  = weight of the specimen after  
drying at 100 to 105 C.

(b) When the percentage of moisture based on the bone-dry weight is required, it shall be calculated as follows:

$$\text{Moisture, per cent} = \frac{W_1 - W_2}{W_2} \times 100$$

where:

$W_1$  = original weight of the specimen,  
and

$W_2$  = weight of the specimen after  
drying at 100 to 105 C.

(c) *Report.*—Report the moisture as



the percentage loss in weight of the specimen to the nearest 0.1 per cent on the basis of the original weight (Paragraph (a)) or dry basis (Paragraph (b)).

#### Reproducibility of Results

6. The results of duplicate determinations of moisture should agree within 0.2 per cent.

## *Standard Method of Test for* OPACITY OF PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 589 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 589; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers the procedure for measuring the opacity of all kinds of paper and paper products by determining the apparent light reflectance.

NOTE.—Other methods, based upon measurements of light transmission, are in general use, but, in the present state of their development, and are not considered to be suitable for standardization.

### Apparatus

2. (a) The apparatus shall be capable of measuring the apparent light reflectance as prescribed in this method. It may measure the values separately, or be designed to give directly the ratio of the apparent reflectances.

(b) Values of apparent light reflectance are relative to the apparent reflectance from magnesium oxide taken as 100 per cent. The standard white backing shall have an apparent reflectance<sup>3</sup> of 91.5 per cent, or an equivalent

cavity may be used. The standard black backing shall have an apparent reflectance of not more than 0.5 per cent.

(c) Completely diffused illumination from incandescent lamps at a color temperature of 2400° to 2800° Kelvin shall be used. The direction of viewing shall be not more than 20 deg. from the normal to the surface of the specimen.

(d) The holder for the specimen shall be so constructed that the specimen is held flat to within 1 per cent of the diameter of the area used for the observation.

(e) Observations shall be made by the human eye or an equivalent means, such as a photoelectric cell with a filter adjusting its sensitivity to that of the human eye.

(f) The instrument shall be calibrated.<sup>4</sup>

### Test Specimens

3. (a) Not less than five specimens

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1944.

<sup>3</sup> See "Preparation and Colorimetric Properties of a Magnesium Oxide Reflectance Standard," *Circular Letter* 393, Nat. Bureau Standards (1934).

<sup>4</sup> Deane B. Judd, "Opacity Standards," *Journal of Research*, Nat. Bureau Standards, Vol. 13, No. 3, p. 281 (1934) (*Research Paper* RP709).

shall be so selected as to be representative of the sample. The shape and size of each specimen will be determined by the equipment used in making the measurements.

(b) The specimens to be measured shall be kept clean and free from folds or wrinkles. The area to be tested shall not be touched with the fingers.

#### Procedure

4. The specimens when tested shall be in equilibrium with the standard atmosphere as prescribed in the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials.<sup>5</sup> Measure the apparent reflectance of the light from the

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

specimen placed over the standard white backing and then over the standard black backing. The ratio of the latter to the former, expressed as a percentage, is the "contrast ratio." It will be nearly 100 per cent for opaque paper; nearly 0 per cent for transparent paper.

#### Calculation

5. The average contrast ratio shall be calculated from at least five determinations.

#### Reproducibility of Results

6. If the mean of the highest and lowest readings differs from the average of all the readings by more than 0.5 per cent, additional specimens shall be tested until there is agreement within this limit.

## *Standard Method of Test for* PARAFFIN CONTENT OF WAXED PAPER<sup>1</sup>



A.S.T.M. Designation: D 590 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 590; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers the procedure for determining quantitatively the amount of paraffin in wax-impregnated (waxed) paper.

NOTE.—The scope of this method is not intended to include paraffin-sized papers. The extract from this type of paper generally contains such quantities of soluble matter other than waxes that the accuracy of the method for small amounts of paraffin is questionable.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Extractor*.—Soxhlet or Underwriters' Laboratories extraction apparatus.

(b) *Funnels*.—Two 250-ml. separatory funnels.

### Reagents

3. (a) *Carbon Tetrachloride, c.p.*

(b) *Petroleum Ether, c.p.*

(c) *Alcoholic Potassium Hydroxide (approximately 0.5 N).*

### Test Specimen

4. The test specimen shall be cut from the sample in such a way as to be thoroughly representative. It shall consist of not less than 3 g. of the paper, in the as-received condition, cut into strips approximately 0.5 in. in width and folded into numerous small crosswise folds (Note).

NOTE.—The folding is essential to secure complete and quick extraction. Do not tear the specimen into small pieces, since they will stick together and the paper may not be completely extracted.

### Procedure

5. (a) Place the prepared test specimen in the extractor, and add carbon tetrachloride ( $\text{CCl}_4$ ). If a Soxhlet extractor is used, the strips of paper should be below the surface of the  $\text{CCl}_4$  when the siphon cup is filled. Extract the specimen until the wax is all removed, collecting the extract in a flask (the extraction generally requires at least 6 hr.). This is not necessary

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

with the Underwriters' Laboratories extractor, where the strips are surrounded by the hot vapors. In either case, enough solvent shall be added to cause the siphon to run over, and then an additional quantity to fill the cup one-half to two-thirds full.

(b) Evaporate the extract on a water bath to dryness, add 25 ml. of the 0.5 *N* alcoholic KOH, and again evaporate to dryness. Cool the dry residue, take it up with petroleum ether and water, and transfer to a separatory funnel. The volume of ether in the funnel shall be about 25 ml. and that of the water 150 ml. The water shall contain a small amount of NaCl to prevent emulsification. Shake the contents of the flask thoroughly, let the two layers separate completely, and draw off the water into a second separatory funnel. Re-extract the water layer one or more times, as may be found necessary, with a fresh 25-ml. portion of petroleum ether. Combine the ether extracts and wash with fresh 100-ml. portions of water until the separated liquids are perfectly clear. An addition of a strong solution of NaCl may be found necessary.

(c) Transfer the petroleum ether extract to a weighed evaporating dish or flask, evaporate to dryness, dry at 100 to 105 C. for 1 hr., and weigh. The weight so found shall be taken to represent the paraffin in the specimen (Note).

NOTE.—The procedure is based on the assumption that all unsaponifiable material soluble in petroleum ether will be paraffin. With the development and growing use of new materials in waxed paper, it may no longer be assumed to contain only paraffin, as there may be present higher alcohols, either free or resulting from saponification of esters or other materials not paraffin, which are soluble in petroleum ether. However, the possible error resulting from these materials is small and may be considered negligible.

#### Report

6. The amount of paraffin shall be reported as a percentage of the original weight of the waxed paper. Not less than two determinations shall be made, and the average result rounded off to the nearest 0.1 per cent shall be reported.

#### Reproducibility of Results

7. Duplicate determinations should agree within 0.2 per cent.

## Standard Method of Test for

### PENTOSANS IN PAPER<sup>1</sup>



A.S.T.M. Designation: D 688 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 688; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. This method of test covers the procedure for determining pentosans in paper.

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Grinder*.—A Koerner<sup>3</sup> or equivalent type grinder that will completely disintegrate the paper without heating or contaminating it.

(b) *Distillation Apparatus*.—A suitable distillation apparatus may be set up as follows: A 300-ml. separatory or dropping funnel is mounted in the neck of a 500-ml. distilling flask, which in turn is connected to a long water-cooled condenser. The condenser delivers through an adapter into a 1-liter reagent bottle, which is used as a receiver and subsequently as a reaction vessel. Rubber stoppers may be used to make the connections. If, during the distillation

(Section 5 (a)), the temperature of the condensate in the receiver is 30 C. or over, owing to inefficient cooling or high laboratory temperature, a small U-trap containing glass beads and 5 ml. of water should be used to close off the system.

#### Reagents

3. (a) *Hydrochloric Acid* (12 per cent, 3.5 N).—Dilute 307 ml. of c.p. HCl (sp. gr. 1.19) to 1 liter.

(b) *Ice*.—The ice used preferably shall be frozen from distilled water. Ordinary commercial ice is satisfactory if uniform values for the blank determinations (Section 5 (c)) are obtained.

(c) *Potassium Iodide Solution* (10 per cent).—Dissolve 10 g. of c.p. KI in 90 g. of water.

(d) *Standard Sodium Thiosulfate Solution* (0.1 N).—Dissolve 25.3 g. of c.p.  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  in water containing 1 ml. of 1 N NaOH and dilute to 1 liter. This gives a solution slightly stronger than 0.1 N, but subsequent calculations are thus made easier. After allowing the solution to stand 1 week, standardize it against 0.1 N  $\text{KMnO}_4$  as follows:

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

<sup>3</sup> The Koerner type of grinder is described in a paper by J. O. Burton and R. H. Rasch on "The Determination of the Alpha-Cellulose Content and Copper Number of Paper," *Journal of Research*, Nat. Bureau Standards, Vol. 6, No. 4, p. 603 (1931) (*Research Paper 295*).

Mix 120 ml. of 6 *N* H<sub>2</sub>SO<sub>4</sub> with 320 ml. of water in a 1-liter, glass-stoppered reagent bottle. Add 10 ml. of KI (10 per cent) and then, after shaking, add 50 ml. of 0.1 *N* KMnO<sub>4</sub> from a pipette. After shaking again, titrate with the thiosulfate solution, adding starch indicator toward the end of the titration.

(e) *Potassium Bromate - Bromide Solution (0.2 N)*.—Dissolve 5.57 g. of c.p. KBrO<sub>3</sub> and 50 g. of c.p. KBr in water. Add 1 ml. of 1 *N* NaOH and dilute to 1 liter. The solution need not be standardized, but blank determinations against the thiosulfate solution shall be made at intervals (Section 5 (c)).

#### Test Specimen

4. The test specimen shall be cut from the sample in such a way as to be thoroughly representative, and shall be reduced to cotton-like form in the disintegrator and then thoroughly mixed.

#### Procedure

5. (a) Allow the specimen to come to moisture equilibrium with the atmosphere of the balance. Weigh, to the nearest milligram, 1 g. of the ground, air-dried specimen for the determination of pentosans. Weigh at the same time specimens for the determination of moisture, ash, and sizing materials. Introduce the specimen into the distilling flask through a dry glass tube so as to avoid adherence of fibers to the neck of the flask. Add 100 ml. of 3.5 *N* HCl, in the neck of the flask and apply heat from a burner. As soon as distillation begins, allow the acid to drip from the funnel at such a rate that the level in the flask remains constant. Continue the distillation at a uniform rate for 100 min., during which period 300 ml. of condensate shall have been collected. It is important to observe this time and quantity.

(b) Add 50 ml. of water to the condensate (if a trap was used (see Section 2 (b))) combine its contents with the condensate and add correspondingly less water), and then add 250 g. of crushed ice. After the temperature of the mixture has fallen to 0 C. or lower, add 20 ml. of 0.2 *N* KBrO<sub>3</sub> - KBr solution from a pipette with a minimum of agitation, close the bottle promptly with a ground-glass stopper, shake well, and allow to stand for exactly 5 min. The temperature should still be 0 C. or lower. Then remove the stopper, add 10 ml. of KI (10 per cent) from a small graduate, and replace the stopper as quickly as possible. Shake the mixture thoroughly to allow absorption of the bromine vapor, and titrate with 0.1 *N* Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

(c) *Blank Determination*.—Make a blank determination in exactly the same manner, using all reagents, including the ice, except that 270 ml. of 3.5 *N* HCl, diluted to 350 ml., shall be used as the starting point, instead of the 300 ml. of condensate diluted with 50 ml. of water.

#### Calculation

6. Calculate the pentosan content as follows:

$$\text{Pentosans, per cent} = \frac{6.80 \times N \times (v_2 - v_1)}{W} - 0.9$$

where:

- N* = normality of the thiosulfate solution,
- v*<sub>1</sub> = milliliters of thiosulfate solution used in the test,
- v*<sub>2</sub> = milliliters of thiosulfate solution used in the blank, and
- W* = weight in grams of the specimen after correcting for moisture, ash, and any filling and sizing materials present.

NOTE 1.—In this method, the furfural formed by the action of hot hydrochloric acid on the fibrous material is distilled from the mixture in essentially the manner prescribed by the Association of Official Agricultural Chemists. The distillates are analyzed for furfural by a rapid volumetric procedure requiring no special apparatus. The volatile material, probably hydroxymethylfurfural, arising from the action of hydrochloric acid upon the cellulose in the samples, is corrected for in a simple manner. The correction, 0.9 per cent, expressed as a pentosan, was found to be valid for the typical chemical wood fibers and papers made therefrom, including unbleached sulfate. For materials containing large quantities of lignin, such as newsprint, the correction is probably not large enough. A correction is also applied for the loss of furfural during the distillation. For further details as to these corrections, or for general information as to the method described, the analyst is referred to the original literature.<sup>4</sup> It is shown therein that the pentosan values for cotton fiber are negligible, being probably less than 0.2 per cent. This point is likewise discussed in the original literature.

<sup>4</sup>H. F. Launer and W. K. Wilson, "Determination of Pentosans in Pulps and Papers," *Journal of Research, Nat. Bureau Standards*, Vol. 22, No. 4, p. 471 (1939) (*Research Paper 1199*).

NOTE 2.—The factor 6.80 is the product of  $100 \times 1.03 \times 0.0480 \div 0.727$ , where 1.03 corrects for a 3 per cent loss of furfural during a distillation, 0.0480 is the weight of furfural in grams corresponding to 1 ml. of 1 *N* thiosulphate solution, and 0.727 is the theoretical conversion factor of pentosans to furfural. If the analyst desires values that are comparable with those obtained by using Krober's tables,<sup>5</sup> in which the conversion of pentosans to furfural is arbitrarily taken as 80 per cent, the factor 6.80 becomes 8.25 and the constant 0.9 (Note 1) is neglected.

### Report

7. The percentage of pentosans shall be reported on the basis of the total cellulose including pentosans, but excluding moisture, ash, fillers, resin, or any sizing or other added nonfibrous materials. All determinations of pentosans shall be made in duplicate, the results of which shall agree within at least 0.4 per cent of the dry weight of the total cellulose content, and the average shall be expressed to the nearest 0.1 per cent.

<sup>5</sup>"Method of Analysis," Assn. Official Agricultural Chemists, Fourth Edition, pp. 641-643 (1935).



## *Standard Method of Test for* PRINTING INK PERMEATION OF PAPER (CASTOR OIL TEST)<sup>1</sup>



A.S.T.M. Designation: D 780 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 780; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method<sup>3</sup> of test covers the procedure for determining the printing ink permeation of paper by measuring the time in seconds in which a drop of U.S.P. castor oil produces a uniform translucent spot through the test specimen. This method is suitable only for easily permeable papers such as news, book, and mimeograph paper. It is a measure of the receptivity of paper to printing inks having an oil vehicle.

### Apparatus

2. The apparatus, as shown in Fig. 1, shall consist of the following:

(a) *Box*.—A box *A*, having an open

front *B*, a ground glass top *C* containing a  $\frac{3}{4}$  in. hole for observation of the specimen *H*, a ground glass partition *D* parallel with the front side to prevent excess heat from affecting the test results, a 25-w. electric bulb *E* placed back of the partition for illumination of the specimen, and an adjustable mirror *F* near the bottom of the box and centered on the hole *G* in the top of the box for observation of the specimen *H*.

(b) *Separatory Funnel*.—A separatory funnel *I* with a tip approximately  $\frac{3}{4}$  in. in length and of such diameter that 25 drops of distilled water delivered at 21 C. (70 F.) will have a volume of 1 ml. The funnel shall be suspended with the end of the tip approximately  $1\frac{3}{4}$  in. above the test specimen and contains U.S.P. castor oil, the temperature of which is maintained during the test at  $25 \pm 2$  C. ( $77 \pm 3.5$  F.).

### Test Specimens

3. (a) The test specimens shall be selected so as to be representative of

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

<sup>3</sup> For further information on this method the following references should be consulted:

E. O. Reed, "Resistance of Printing Papers," *Paper Trade Journal*, Vol. 86, No. 12, March 22, 1928, p. 54; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 11, No. 1, pp. 144-145 and 239-240 (1928).

B. L. Wehmhoff, "Evaluation of the Printing Quality of Paper," *Paper Trade Journal*, Vol. 100, No. 6, February 7, 1935, p. 41; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 18, No. 55, June, 1935, pp. 337-340.

the sample secured by the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D

585).<sup>4</sup> Any convenient size specimen may be used, preferably 2-in. square. All specimens shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>4</sup>

#### Procedure

4. (a) The test shall be made in standard atmospheric conditions as specified in A.S.T.M. Method D 685.

(b) The test specimens shall be placed over the hole G in the top of the box as shown in Fig. 1. A drop of U.S.P. castor oil shall be allowed to fall from the funnel upon the specimen and a stop watch shall be started the instant the drop strikes the specimen. The under side of the specimen shall then be observed and the time interval measured from the instant of contact of the oil with the paper until the spot of oil reaches a uniform and maximum translucency. The spot of oil shall be covered with a cap having a black interior which aids in the determination of the end point.

#### Report

5. The report should give the maximum, minimum, and average time of penetration to the nearest 5 sec. for not less than five tests on each side of the paper.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

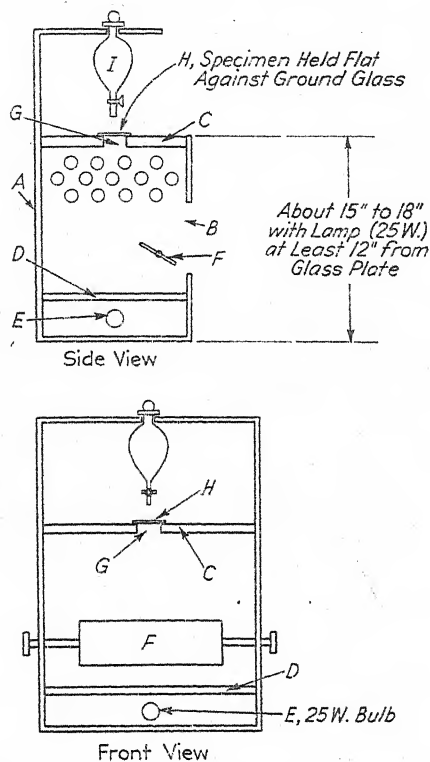


FIG. 1.—Apparatus for Printing Ink Permeation Test of Paper.

- A—Box.
- B—Open front in box.
- C—Top, ground-glass.
- D—Partition, ground-glass.
- E—Electric bulb, 25 w.
- F—Adjustable mirror.
- G—Hole in top of box for observation of specimen.
- H—Test specimen.

## *Standard Method of Test for* ROSIN IN PAPER AND PAPERBOARD<sup>1</sup>



A.S.T.M. Designation: D 549 - 46

ADOPTED, 1941; REVISED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 549; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method covers the procedures for qualitative and quantitative determination of rosin (colophony) in paper and paperboard.<sup>3</sup> The qualitative tests indicate the presence or absence of resin from rosin or rosin soap added as a sizing agent. The quantitative tests give the total rosin which consists of the natural resins in the pulp from which the paper is made plus any rosin added as sizing. Such procedures are not applicable to the determination of synthetic resins (such as the melamine and urea-formaldehyde resins used to impart wet strength properties to paper). The method is limited to rosin, which is a natural vegetable resin, the residue left after

distilling off the turpentine from the gum of pine or from the solvent extraction of pine stumps, knots and top wood with subsequent separation from the extraction solvent, turpentine or pine oil, by distillation.

### QUALITATIVE TEST

#### Qualitative Test

2. A positive result obtained by both of the procedures described in Paragraphs (a) and (b) shall be regarded as conclusive evidence of the presence of rosin or rosin soap added as a sizing material.

(a) *Liebermann-Storch Test*.—Place about 1 g. of the paper, cut into small pieces, in a clean, dry test tube. Add 5 ml. of c.p. acetic anhydride and evaporate by boiling to about 1 ml. (*Caution*: The fumes of the anhydride are very irritating and should be burned as they leave the mouth of the test tube.) Pour the liquid residue into a clean, dry porcelain crucible and cool to room temperature. If any waxy particles separate out, filter them off on a dry filter paper. Add carefully, down the side

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

For further information on this method the following references may be consulted:

C. F. Sammet, "Quantitative Determination of Rosin in Paper," *Industrial and Engineering Chemistry*, Vol. 5 p. 732, September (1913); Paper 13, No. 1, 17 (September, 1913).

E. Sutermeister and W. V. Torrey, "The Determination of Rosin in Paper," *Paper Trade Journal*, Vol. 94, No. 3, p. 33, January 21, 1932.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

<sup>3</sup> For further information, see "Paper and Paperboard, Characteristics, Nomenclature and Significance of Tests," p. 78, Am. Soc. Testing Mats. (1944). (Monograph issued as separate publication.)

of the crucible, one drop of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84). A fugitive, rose-violet coloration formed where the acid meets the anhydride indicates rosin.

(b) *Raspail Test*.—Place the paper on a glass or porcelain plate and apply a drop of a nearly saturated solution of sugar. After a few moments remove the excess sugar solution with filter paper. Add a drop of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) to the sugar on the paper. A raspberry-red coloration indicates the presence of rosin.

#### QUANTITATIVE DETERMINATION

NOTE.—This procedure for the quantitative determination of rosin depends upon the conversion of insoluble resins to alcohol-soluble rosin by the action of a strong volatile acid of a definite concentration for a definite length of time. Thus, a minimum amount of nonresinous materials, such as cellulose hydrolysis products, is formed. Separation of the resinous from the nonresinous materials is accomplished by dissolving out the rosin from the dried extract with anhydrous ether, in which nonresinous materials, including starches and glues, are insoluble. For further details consult the original literature.<sup>4</sup> If waxes and fats that may dissolve in the anhydrous ether are present, they must be separated from the resinous material by saponification.

If the presence or absence of mineral matter which reacts with hydrochloric acid or is dissolved by it, and of paraffin or similar wax, in the paper under test is not definitely known, qualitative tests for them should be made, as their respective absences permit the omission of certain steps in the procedure.

#### Apparatus

3. A suitable extraction apparatus, such as the Soxhlet or Underwriters' Laboratories flask, is required.

#### Reagent

4. *Extraction Solvent*.—Add 4 ml. of  $\text{HCl}$  (sp. gr. 1.19) to 1 liter of 95 per cent ethyl alcohol and mix.

#### Test Specimen

5. The test specimen shall consist of strips about 0.25 by 1.6 in. (6 by 40 mm.). The strips shall be cut from the sample in such a way as to be thoroughly representative. The specimen should not be ground, for more nonresinous materials are then extracted.

#### Procedure

6. (a) With the test specimen in moisture equilibrium with the atmosphere of the balance, weigh out 5 to 7 g. of the strips to the nearest 10 mg. Immediately thereafter, weigh out test specimens for the moisture determination (Note 1), which shall be made in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (A.S.T.M. Designation: D 644).<sup>5</sup>

NOTE 1.—If the paper contains paraffin or similar wax, or oil, it may not be practicable to determine moisture. In such cases the moisture determination may be omitted.

(b) If mineral matter reacting with, or dissolved by, hydrochloric acid is present, immerse the test specimen for the rosin determination in approximately normal aqueous  $\text{HCl}$  for 5 min., drain off the acid solution, wash the specimen free from acid with tap water followed by distilled water, and dry at room temperature. If acid-reactive or acid-soluble mineral matter is absent, omit this preliminary acid treatment.

(c) Place the strips lengthwise in the siphon cup of the extractor, taking care to avoid packing them together tightly. For the extraction use about two to two and one-half times the volume of solvent required to fill the siphon cup. Extract at a rate of 15 siphonings per hour (which should yield a volume of about 250 ml. of solvent distilled per hour) for a period of 2 hr. for uncoated or non-surface-sized papers and 2.5 hr.

<sup>4</sup> H. F. Launer, "Simplified Determination of Resin in Papers and Pulps," *Journal of Research*, Nat. Bureau Standards, Vol. 18, No. 2 (February, 1937) (*Research Paper 973*).

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

for coated or surface-sized papers (Note 2). The extraction should not be continued much longer than specified.

NOTE 2.—The time of extraction specified may not be sufficient for certain heavy types of paperboard, such as container board, press-board, binders board and pasted board, and for such paperboards the extraction procedure may not yield strictly quantitative results.

(d) When the extraction period is completed, evaporate the solvent in the flask on the steam bath until the odors of alcohol and HCl are no longer noticeable. Place the flask in an oven at 100 C. for 15 min., cool to room temperature, and add 20 ml. of anhydrous ether. The rosin dissolves in from 5 to 30 sec., but if covered by foreign material it must be uncovered by scraping the mass with a stirring rod. If necessary, let the ether solution stand 15 to 20 min. to further the coagulation and the settling out of the foreign matter. Filter the solution with suction through a filter paper of close texture, cut to fit a Gooch crucible, directly into a weighed beaker. It is usually necessary to refilter the filtered solution through the same paper. Rinse with another 20-ml. portion of ether, adding the rinsing to the solution in the weighed container. Evaporate the ether, then dry in an oven at 100 C. for 15 min. and weigh to the nearest 1 mg. Repeat the drying and weighing until the weight is constant to within plus or minus 1 mg. (Note 3).

NOTE 3.—There is a possibility that there may be present materials other than rosin which might be extracted and therefore would be reported as rosin.

(e) If paraffin or similar waxes are present, after weighing the container plus rosin and wax add about 25 ml. of 0.5 N alcoholic potash solution, heat to not over 60 C. for 15 min., cool to room temperature, and transfer to a separatory funnel. Wash the beaker with about 25 ml. of ether and then with

about 50 ml. of water, adding all washings to the separatory funnel. Add sufficient water or ether, or both, so that the funnel contains about 25 ml. of ether and 150 ml. of water. Shake the funnel well, add about 2 g. of NaCl, shake the funnel once more, and let stand until the liquids have separated. Draw off the water solution into another separatory funnel and wash with 25 ml. of ether. Drain the two ether solutions into a weighed beaker, wash both funnels with about 20 ml. of ether, adding the washings to the beaker, and evaporate the ether. Dry the container and residue at 100 C. (Note 4) for 15 min. and weigh to the nearest 1 mg. Repeat the drying and weighing until weight is constant within plus or minus 1 mg. The material thus obtained will be paraffin or similar wax, plus the unsaponifiable material from the rosin, which, for most purposes and in the absence of definite knowledge regarding the particular rosin in the paper under test, may be assumed to be 5 per cent of the rosin in the paper. To obtain the weight of the rosin, divide the weight of the combined rosin and wax, less the weight of the wax, by 0.95.

NOTE 4.—If the paper contains waxes or oil volatile at 100 C. and it is not practicable to dry the residue at a lower temperature, the rosin should be separated from the oil by saponification, and determined directly, as prescribed in the following alternate method.

(f) *Alternate Treatment of Rosin - Wax Extract Where a Direct Determination of Rosin is Desired.*—Wash the weighed rosin - wax residue obtained from the alcohol-extracted paper into a wide-mouthed flask, using about 25 ml. of ether. Add about 25 ml. of 0.5 N alcoholic potash and about 100 ml. of water. Heat the contents of the flask to boiling under a reflux condenser for

30 min., cool to room temperature, add about 25 ml. of ether, transfer to a separatory funnel, and wash the flask first with water and then with ether, adding the washings to the funnel. Add about 2 g. of NaCl to the separatory funnel, shake well, and let stand until the liquids have separated. Draw off the water solution into a beaker. Add 50 ml. of water and about 2 g. of NaCl to the ether solution in the separatory funnel, shake well, let stand until the liquids have separated, and draw off the water solution, adding it to the first one obtained. Add to the combined water solutions 2 drops of methyl orange indicator, then carefully acidify by adding 5 *N* H<sub>2</sub>SO<sub>4</sub> until the solution is definitely pink, and then add 1 ml. more of the acid. Cool the solution to room temperature, transfer to a separatory funnel, add 25 ml. of ether, and wash the beaker with about 10 ml. of ether, adding the washings to the funnel. Add about 5 g. of NaCl, shake the funnel well, let stand until the liquids have separated, draw off the water solution into another separatory funnel, and wash it twice more, using about 20 ml. of ether each time. Transfer all ether solutions to a weighed

beaker, washing all containers with ether. Evaporate the ether, dry in an oven at 100 C. for 1 hr. and weigh to the nearest 1 mg. Repeat the drying and weighing until weight is constant to within plus or minus 1 mg. The weight of the rosin thus found is assumed to be 95 per cent of that in the paper.

NOTE 5.—If the paper contains saponifiable fats or greases in addition to rosin, the alternate method described above will give results higher than the true rosin content, as they will be included with it. In this case, the analyst may apply such modifications to the method as are considered justifiable and the report should state what modifications of the method were used.

#### Report

7. The result of the rosin determination shall be expressed to the nearest 0.1 per cent by weight of the paper dried at 100 to 105 C. whenever practicable. When this is not practicable, it shall be expressed as a percentage of the paper as received or air-dry and the report shall so state.

#### Reproducibility of Results

8. The report shall be the average of at least two determinations. The percentage results of duplicate determinations should agree within 0.2.

## *Standard Method of* SAMPLING PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 585 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 585; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method covers the procedure for sampling paper and paper products.

### Sample

2. (a) The sample, unless otherwise specified, shall consist when possible of specimens each cut not less than 11 by 11 in. This allows margin for trimming to exactly 10 by 10 in., which simplifies the calculation in the basis weight determination. A sufficient number of specimens to complete the tests shall be taken.

(b) The specimens shall be kept smooth and flat, and protected from exposure to direct sunlight, contact with liquids, and other harmful influences. Care shall be exercised in handling the specimens if acidity or other chemical characteristics, optical, surface or other physical characteristics affected by the moisture of the hands are to be determined. Specimens to be tested for

moisture shall be placed immediately after sampling in an airtight container for storage.

### Size of Sample

3. The specimens comprising the sample shall be so selected as to be representative of the entire lot of paper. Not less than 5 nor more than 20 sets of specimens, comprising one set from each unit, shall be taken as follows:

Total Units in Shipment	Units to be Sampled
Less than 100.....	5
100 to 399.....	5 to 20 <sup>a</sup>
400 or more.....	20

<sup>a</sup> That is, 5 per cent of the total number of units in the shipment.

The units shall be rolls, cases, frames, skids, or bundles.

### Method of Sampling

4. (a) In the case of rolls, care shall be taken to select sheets that are not damaged. It is good practice to discard the first three layers of the roll to be sure of obtaining a unit sample in good representative condition. The

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

specimens shall be cut from sheets taken across the full width of several unharmed layers.

(b) In the case of sheet-cut paper, specimens shall be cut from at least five consecutive sheets taken from a point or points over  $\frac{1}{2}$  in. from the top or bottom of each case, frame, skid, or bundle.

(c) The specimens shall be trimmed with their edges exactly parallel to the machine and cross directions of the paper.

(d) A sufficient number of specimens from each unit sampled shall then be arranged consecutively in rotation to form a representative sample.

#### Resampling

5. In case of necessity for resampling a lot of paper, the samples shall be taken as prescribed in Sections 2 to 4, except that they shall be taken from different units than those previously sampled.

NOTE.—Physical tests, except for weight, shall not be made on portions of specimens in which there are flaws or watermarks.



## Standard Method of Test for STARCH IN PAPER<sup>1</sup>



A.S.T.M. Designation: D 591 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 591; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers procedures for the qualitative and quantitative determination of starch in paper.

### QUALITATIVE TEST

#### Qualitative Test

2. A positive result obtained by the following procedure shall be regarded as conclusive evidence of the presence of starch in paper:

Boil about 0.5 g. of the sample for several minutes with 10 ml. of water. Filter off the extract, cool it, and add one drop of approximately 0.001 *N* iodine solution (Note). A blue coloration indicates starch. If only a faint violet coloration is obtained, this should be disregarded, as constituents of paper other than starch sometimes give such a reaction.

NOTE.—It is convenient to prepare an approximately 0.01 *N* stock solution of iodine

(0.13 g. of iodine and 2.6 g. of KI in 100 ml. of water) and dilute a portion of this to a pale yellow color (about 0.001 *N*) each time a test for starch is made.

### QUANTITATIVE DETERMINATION

#### Apparatus

3. *Balance*.—The balance shall be sensitive to 1 mg.

#### Reagents

4. (a) *Ammonium Hydroxide* (approximately 6 *N*).—Mix 400 ml. of  $\text{NH}_4\text{OH}$  (sp. gr. 0.90) with 700 ml. of water.

(b) *Hydrochloric Acid* (5:4).—Dilute five volumes of c.p.  $\text{HCl}$  (sp. gr. 1.19) with four volumes of water.

(c) *Iodine Solution* (approximately 0.02 *N*).—Dissolve 5 g. of KI in about 10 ml. of water; then dissolve in this solution 2.6 g. of iodine and dilute to 1 liter.

(d) *Universal Indicator*.<sup>3</sup>—Dissolve 0.005 g. of thymol blue, 0.0125 g. of

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1940 to 1942.

<sup>3</sup> S. Yamada, Japanese Patent 99664, February 21, 1933; *Chemical Abstracts*, Vol. 28, April 20, 1934, p. 2258; D. T. Jackson and John L. A. Parsons, "A Universal Hydrogen Ion Indicator," *Tech. Assoc. of Pulp and Paper Industry*, Papers 1936, p. 236.

methy1 red, 0.050 g. of bromthymol blue, and 0.100 g. of phenolphthalein in 100 ml. of neutral, 95 per cent ethyl alcohol. Neutralize the solution with 0.05 *N* NaOH and dilute to 200 ml. with water. The indicator solution should then be green.

To determine the pH of a solution, add 0.5 ml. of the universal indicator to 5 ml. of the solution under test in a comparator tube approximately 0.5 in. in inside diameter, mix, and note the color of the mixture. The color indications for pH values from 4 to 10 are as follows:

Color	pH Value
Red.....	4
Orange.....	5
Yellow.....	6
Green <sup>a</sup> .....	7
Blue <sup>b</sup> .....	8
Indigo Blue.....	9
Violet.....	10

<sup>a</sup> Light green with solutions obtained from paper.

<sup>b</sup> Dark green with solutions obtained from paper.

NOTE.—The universal indicator described is that of Yamada.<sup>3</sup> It is useful for determining the pH value of a solution to the nearest whole number.

(e) *Asbestos for Gooch Filters*.—Digest the asbestos, which should be the amphibole variety, with diluted HCl (1:3) for 2 to 3 days at room temperature. Wash free of acid. Digest for a similar period with NaOH solution (10 per cent), and then treat for 3 to 4 hr. with hot, alkaline tartrate solution of the strength used in sugar determinations (solutions that have stood for some time may be used). Wash the asbestos free of alkali, digest with diluted HNO<sub>3</sub> (1:3) for 3 to 4 hr., wash free of acid, and shake with water into a fine pulp.

(f) *Fehling's Solution (Allihn Modification)*:

*Solution A*.—Dissolve 69.3 g. of CuSO<sub>4</sub>·5H<sub>2</sub>O, of high purity and as free from Fe as possible, in 1 liter of water.

*Solution B*.—Dissolve 250 g. of KOH of good quality and 345 g. of Rochelle

salts (potassium sodium tartrate) in 1 liter of water.

Mix equal volumes of solutions A and B immediately before use.

(g) *Phosphomolybdate Solution*.—Dissolve 100 g. of sodium molybdate (43 per cent molybdenum) and 75 ml. of syrupy H<sub>3</sub>PO<sub>4</sub> (85 per cent) in a solution consisting of 275 ml. of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) added to 1750 ml. of water.

(h) *Potassium Permanganate Solution* ( $\frac{1}{10}$  *N*).—Dissolve about 16 g. of KMnO<sub>4</sub> in 1 liter of water. Allow this approximately 1 *N* solution to stand in the dark, preferably for one month, then dilute a portion of it thirty times and standardize against the National Bureau of Standards standard sample 40c of sodium oxalate.

### Test Specimen

5. (a) The test specimen shall consist of 5 g. of paper, weighed to the nearest 5 mg. It shall be thoroughly disintegrated (Note). Soft papers may be macerated in a small quantity of water. Hard papers may have to be torn into small pieces and shaken in a stoppered bottle with about 75 ml. of water and some lead shot, after which the lead shot shall be removed. However prepared, the test specimen shall be thoroughly representative of the sample of paper submitted for test.

NOTE.—The paper should not be ground, as papers containing mineral filler, when disintegrated in a mechanical grinder, lose considerable of the mineral filler and may also lose starch.

(b) At the time the specimen for the starch determination is weighed, a separate test specimen shall be prepared and tested for moisture in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (A.S.T.M. Designation: D 644) of the American Society for Testing Materials.<sup>4</sup>

<sup>4</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

**Procedure<sup>5</sup>**

6. (a) If the test specimen is acidic, dilute it to 100 ml., heat to boiling, and then add two drops of 6*N*  $\text{NH}_4\text{OH}$ . This should bring the pH value of the solution to 7 and the high temperature should tend to vaporize any excess of  $\text{NH}_3$ . If the pH value of the solution is not 7, adjust it to 7 as measured with the universal indicator. If the mixture is alkaline, add 5 ml. of  $\text{HCl}$  (5:4) and then neutralize the excess acid with a slight excess of  $\text{NH}_4\text{OH}$ . Expel the slight excess of the latter by boiling as before, after dilution to 100 ml.

(b) To 100 ml. of water add 0.5 g. of  $\text{NaCl}$ . After washing out the mouth thoroughly with water, collect 10 ml. of saliva (Note 1), add to the salt solution, and heat to 50 C.

(c) Cool the water suspension of the test specimen to 50 C., and add the saliva-salt solution. Store the mixture at 40 to 55 C. for 1 to 4 hr., then remove a few fibers and test for unconverted starch with 0.02 *N* iodine solution. If a blue coloration appears, filter the main solution and digest the pulp with another 10 ml. of saliva as before (Note 2). Test the pulp again for unconverted starch. Continue the digestions with saliva-salt solution until the pulp no longer gives a blue color with 0.02 *N* iodine solution.

NOTE 1.—Some salivas are more active than others, and therefore it may not always be necessary to use 10 ml. Some salivas may also show a slight reducing reaction with Fehling's solution. It is advisable to test the saliva for such effect; if found, run a blank test and apply the proper correction to the final test results. However, this condition will only rarely be found if the saliva is properly collected.

<sup>5</sup> See R. C. Griffin, "Technical Methods of Analysis," Second Edition, McGraw-Hill Book Co., Inc., New York, N. Y., (1927), p. 479; also W. Totttingham and M. Gerhardt, "Quantitative Methods for the Analysis of Hemicellulose in Apple Wood," *Industrial and Engineering Chemistry*, Vol. 16, p. 139 (1924).

NOTE 2.—The second digestion with saliva is usually necessary where fairly large amounts of rosin sizing are used, or the paper has been overdried. If the stock has been very thoroughly beaten, or the paper overdried, the pulp, when tested after the digestion with saliva-salt solution, may persistently give a faint blue color. This, however, is not a positive indication of unconverted starch, but indicates that the cellulose has attained the same iodine dispersing power as the starch. Few papers require more than two digestions with saliva.

(d) Filter the solution through a Büchner funnel, being careful that no fibers reach the filtrate, and after sucking all the water through the funnel, wash the pulp three times with boiling water, draining completely after each washing. Combine all filtrates, add a few drops of  $\text{HCl}$ , and concentrate to 200 ml. by evaporation on the steam bath. Transfer to an Erlenmeyer flask, add 20 ml. of  $\text{HCl}$  (5:4), fit the flask with a reflux condenser, and boil moderately for 2 hr. Cool the solution and transfer to a 250-ml. volumetric flask. After diluting to the mark and mixing, pipette 25 ml. of the solution into a 400-ml. beaker, and slowly add dry  $\text{Na}_2\text{CO}_3$ , while stirring, until effervescence ceases.

(e) To 30 ml. of Fehling's solution A in a 250-ml. beaker, add 30 ml. of solution B and 60 ml. of water. Heat to boiling, making sure no visible reduction takes place, add to the solution from the paper, and boil for exactly 2 min., keeping the beaker covered. Filter at once without dilution, and determine the amount of  $\text{Cu}_2\text{O}$  formed, either gravimetrically or volumetrically as described in Paragraph (f) or (g).

(f) *Gravimetric Method*.—Prepare a Gooch crucible with a pad of the special asbestos, approximately  $\frac{1}{2}$  in. in thickness. Wash thoroughly with water by suction to remove fine particles; then wash with 10 to 15 ml. of alcohol, and finally with 10 to 15 ml. of ether. Dry in an oven at 100 to 105 C. for 30 min.,

cool in a desiccator, and weigh. Decant the major portion of the solution containing the precipitated  $\text{Cu}_2\text{O}$  through the Gooch crucible under suction; then transfer the precipitated oxide, and wash five times with hot water, three times with alcohol, and three times with ether. Dry the crucible and contents at 100 to 105 C. for 30 min., and weigh the  $\text{Cu}_2\text{O}$ . From the weight of  $\text{Cu}_2\text{O}$ , calculate the amount of dextrose according to the tables of Allihn.<sup>6</sup> The calculated amount of dextrose multiplied by 0.9 gives the amount of starch in the aliquot part of the solution.

(g) *Volumetric Method*.—Filter the solution containing the  $\text{Cu}_2\text{O}$  through asbestos in a small Büchner funnel and wash with about 200 ml. of hot water. Transfer the asbestos mat and the  $\text{Cu}_2\text{O}$  back to the precipitation beaker, add 25 ml. of the phosphomolybdate solution, and mix thoroughly with a glass stirring rod. Filter through the same Büchner funnel, and wash thoroughly

with cold water until all trace of blue color is removed. Dilute the filtrate to 400 to 700 ml., and titrate with  $\frac{1}{30} N$   $\text{KMnO}_4$  to a faint pink color after the blue color has been discharged. If the titration requires less than 15 ml. of  $\frac{1}{30} N$   $\text{KMnO}_4$ , the analysis should be repeated, using a 50-ml. aliquot of the prepared solution from the paper (Paragraph (d)), and using 35 ml. instead of 60 ml. of added water (Paragraph (e)).

$$\begin{aligned} 1 \text{ ml. of } \frac{1}{30} N \text{ KMnO}_4 &= 0.0011 \text{ g. of} \\ \text{dextrose} &= 0.00099 \text{ g. of starch} \end{aligned}$$

NOTE.—Calculation of the weight of  $\text{Cu}_2\text{O}$  is not necessary if an accuracy of 0.1 to 0.2 per cent starch, dry-paper basis, is sufficient. If desired, the weight of  $\text{Cu}_2\text{O}$  may be calculated from the volume of  $\text{KMnO}_4$ , and the tables of Allihn<sup>6</sup> may be consulted for the corresponding weight of dextrose. This may not be more accurate, however, than using the factor given, since the various kinds and grades of starch differ as to relationship to  $\text{Cu}_2\text{O}$ . If possible, the factor should be determined for the kind of starch contained in the paper.

### Report

7. The amount of starch shall be reported as a percentage of the moisture-free paper to the nearest 0.1 per cent.

<sup>6</sup> See A. E. Leach, "Food Inspection and Analysis," Fourth Edition, John Wiley and Son, New York, N. Y. (1920), p. 633; also "Methods of Analysis," Assn. of Official Agric. Chemists, 1935, pp. 636 and 637.

## *Standard Method of Test for* SURFACE WETTABILITY OF PAPER (ANGLE-OF-CONTACT METHOD)<sup>1</sup>



A.S.T.M. Designation: D 724 - 45

ADOPTED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 724; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) This method<sup>3</sup> of test is intended for the quantitative determination of the resistance of paper surfaces to wetting. It is useful for the determination of writing and ruling qualities of papers, as well as for other problems related to the resistance which the surface of paper offers to water and aqueous vehicles. This method deals with the behavior of a drop of water, or liquid solution, when applied directly to the surface.

(b) The procedure described in this method involves the measurement of the angle of contact, since the advancing angle of contact between a liquid and a smooth solid surface is a measure of the wettability of the latter by the former.

### Apparatus

2. (a) *Preferred Apparatus.* — The preferred projection apparatus, a side elevation of which is shown in Fig. 1, shall consist of the following:

(1) *Lamp.* — A ventilated lamp house containing a 250-w. projection lamp.

(2) *Tube and Lens.* — A tube containing a lens to concentrate the beam of light.

(3) *Water Cell.* — A water cell inserted between the source of light and the lens for concentrating the light rays on the stage on which the specimen is placed.

(4) *Stage.* — A horizontal stage to hold the test specimen, with means for vertical adjustment of the stage.

(5) *Microscope.* — A microscope draw tube fitted with a 25-mm. objective and a 5× ocular mounted horizontally beside the stage.

(6) *Hypodermic Syringe.* — A syringe, such as a 1-ml. hypodermic, equipped with a No. 27 stainless steel

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1943 to 1945.

<sup>3</sup> For further information on this method the following reference may be consulted:

G. H. Lafontaine, "Surface Wettability of Paper and the Angle of Contact Test," *Paper Trade Journal*, Vol. 113, No. 6, p. 29 (August 7, 1941).

needle, capable of providing 150 to 200 drops per milliliter.

(7) *Screen*.—A frosted glass screen with clamps to hold a sheet of paper.

(b) *Alternate Apparatus*.—If the apparatus described in Paragraph (a) is not available, a microscope may be used for projecting the image of the drop on the glass screen. A small projection microscope with a magnification of about 30 $\times$  is suitable for this purpose. A small burette or pipette adjusted to deliver 1/150 to 1/200 ml. may be substituted for the hypodermic syringe.

dyes shall not be used. The ink shall be prepared as follows: Dissolve the tannic acid and gallic acid in about 400 ml. of water at about 50 C. (122 F.). In a separate vessel, dissolve the ferrous sulfate in about 200 ml. of water which contains the hydrochloric acid. In a third vessel, dissolve the dye in about 200 ml. of water. Mix the three solutions in a 1-liter volumetric flask, rinse the vessels with small portions of water, and add the rinsings to the flask. Add the carbolic acid. Dilute with water to 1 liter at 20 C. (68 F.).

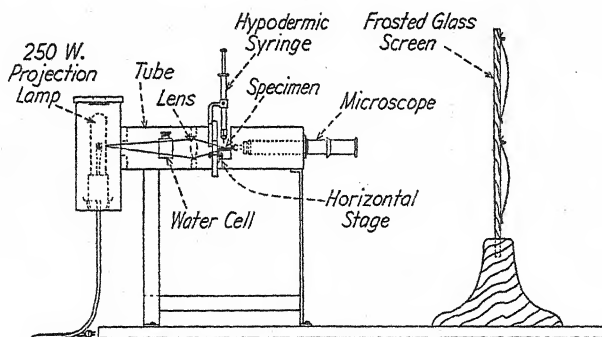


FIG. 1.—Preferred Projection Apparatus.

### Test Liquids

3. (a) *Standard Ink*.—A standard ink having the following composition shall be used for determining the writing qualities of the paper:

Tannic acid, g.....	11.7
Gallic acid crystals, g.....	3.8
Ferrous sulfate, g.....	15.0
Hydrochloric acid, dilute, g....	12.5
Carbolic acid (phenol), g.....	1.0
Soluble blue (Shultz No. 539, Color Index 707), g.....	3.5
Water.....	to make a volume of 1000 ml. at 20 C. (68 F.)

All chemicals used in preparing the ink shall be of c.p. quality. Some blue dyes react with carbolic acid to cause a film having a metallic appearance and such

(b) *Ruling Fluid*.—A solution of 0.01 g. of a water-soluble blue dye dissolved in 100 ml. of distilled water shall be used for determining the ruling qualities of the paper.

(c) *Water*.—For determining surface wettability, distilled water is preferable, but water of ordinary purity (tap water) may be used.

### Sampling

4. The paper shall be sampled in accordance with the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>4</sup>

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Test Specimens

5. (a) The test specimens shall consist of strips  $\frac{1}{2}$  in. in width and 4 in. in length, cut from the paper sample in such a way as to be thoroughly representative of it. Tests shall be made on both sides of each specimen. The actual areas tested shall not contain watermarks or visible blemishes or defects, and shall not be touched with the fingers or contaminated in any other way.

(b) The test specimen shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials.<sup>4</sup>

### Procedure

6. (a) Test the specimens under standard atmospheric conditions as prescribed in Method D 685.

(b) Place a test specimen on a slide on the stage and hold it in close contact with the slide by means of small weights placed close enough on each side of the needle to prevent distortion of the specimen when it is wetted by the drop of test liquid.

(c) Set the tip of the hypodermic needle  $\frac{1}{8}$  in. from the surface of the specimen and deposit a drop of the test liquid 1/150 to 1/200 ml. in size on the specimen. Project the image of the drop, enlarged 25 to 30 times, on the glass screen, at the back of which shall be clamped a sheet of transparent onionskin paper. Draw a horizontal line on the transparent paper coinciding with the image of the base of the drop and as soon as the specified time of contact of the drop with the specimen has elapsed, quickly draw two tangents to the curve at the two points of contact with the base line. The average of the interior angles between the base line and the two tangents is designated as the angle of

contact. Measure the angles with a protractor.

(d) For initial wettability, used as a measure of ruling quality, make the measurement of the angle of contact after the drop has been in contact with the paper for 5 sec. For rate of change in wettability, used as a measure of writing quality, make two measurements, one after 5 sec. and the other after 60 sec. have elapsed.

(e) Make measurements for 5 drops on each side of the specimen. Between successive tests, move the transparent paper on the screen to a new position to avoid confusion of lines.

### Calculation and Report

7. (a) *Calculation.*—Calculate the rate of change in wettability as follows:

$$R = \left( \frac{A - a}{55} \right)$$

where:

$R$  = rate of change in wettability in degrees per second,

$A$  = average angle of contact after 5 sec., and

$a$  = average angle of contact after 60 sec.

(b) *Report.*—The report shall include the following:

(1) The initial wettability reported as the average angle of contact after 5 min. exposure, expressed to the nearest degree, or rate of change in wettability calculated in accordance with Paragraph (a) and reported to two significant figures for each side of the specimen. If possible to identify the two sides, the results shall be identified as to the wire side and the felt side; otherwise, the sides may be arbitrarily designated, as for example, A and B.

(2) Maximum, minimum, and average test results for each side of the specimen tested, reported to two significant figures.

(3) The test liquid used.

### Reproducibility

8. While variations between individual drops on one side of a specimen may be as great as 5 deg., the average of 10 angles of contact will usually not vary by more than 2 deg. between two specimens cut from the same sheet of paper, when tested on the same side of the sheet, unless the variations in finish between the two specimens cut from different parts of the sheet are too pronounced.

### Interpretation of Results

9. (a) In considering the results of the angle-of-contact test, the following factors which all affect the results in different degrees shall be considered: The wetting power of the test liquid used, the wettability of the sizing agent used in sizing the paper, and the surface texture or finish of the paper.

(b) The standard ink will give smaller angles of contact than water, indicating that it wets the paper more readily than water. Papers surface-sized with starch will generally show smaller angles than papers of about the same finish tub-sized with glue. A machine-finished paper with a grainy surface would have a greater angle of contact than a plated or calendered paper of equal sizing. It is known that, in practice, ruling results

will depend on both the surface wettability and finish of the paper. Thus, the angle of contact should give a very good idea of what is to be expected. It has been found that excellent ruling will prevail when the average angle of contact with water lies between 100 and 90 deg.; when the angle of contact is greater than 110 deg., breaks are likely to occur in the ruled lines; when the angle is smaller than 90 deg., the ruling fluid is likely to feather.

(c) In determining the writing qualities of paper by means of the angle-of-contact method, it is necessary to take into account that medium-sized papers will at times show feathering only after the ink has partly penetrated the paper. The tendency of a writing paper to feather will be indicated by the decrease in the angle of contact between measurement after 5 and 60 sec. In hard-sized papers, the angle of contact will not change perceptibly between the measurements at 5 and 60 sec. If the initial wettability is less than 90 deg., it is quite likely the paper will feather as soon as it is written upon.

(d) As the range in contact angle for specific conditions is small, refined technique is required in performing the test. This is indicated by the above examples as well as by theoretical considerations.



## *Standard Method of Test for* INTERNAL TEARING RESISTANCE OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 689 - 44

ADOPTED, 1944.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 689; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method covers the procedure for determining the average force in grams required to tear a specimen of paper.

### Apparatus

2. (a) The testing machine shall be of the pendulum impulse type and shall consist of the following:

(1) A stationary clamp,

(2) A movable clamp carried on a pendulum, preferably formed by a sector of a wheel or circle, free to swing on a ball bearing or other substantially frictionless bearing,

(3) Means for holding the pendulum in a raised position and means for releasing it instantaneously, and

(4) Means for registering the maximum arc through which the pendulum swings when so released. The pendulum shall carry a circumferential scale, graduated from 0 to 100 so as to read against the pointer the work done in gram-centimeters by the pendulum when

tearing a specimen of paper, divided by 137.6 (one-sixteenth of the force in grams required to tear a specimen) (Note 1).

(b) With the pendulum in its initial position ready for a test, the two clamps shall be separated by an interval of 2.5 mm. (0.10 in.) and so aligned that the specimen clamped in them lies in a plane perpendicular to the plane of oscillation of the pendulum with the edges of the jaws gripping the paper in a horizontal line, a perpendicular to which through the axis of suspension of the pendulum is 104 mm. (4 in.) in length and makes an angle of 27.5 deg. with the plane of the paper.

(c) The clamping surface in each jaw shall be over 25 mm. (1 in.) in width and over 12 mm. (0.5 in.) in depth.

### Adjustment of Apparatus

3. (a) For apparatus having a sector as a pendulum, and which tears the specimen as it moves toward the right, draw a pencil line on the base or stop-mechanism 1 in. to the right of the edge of the sector stop. With the sector raised to its initial position and the pointer set against its stop, on releasing

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1942 to 1944.

the sector and holding the stop down, the sector should make at least 20 complete oscillations before the edge of the sector which engages with the stop no longer passes to the left of the pencil line. Otherwise, the bearing shall be oiled and adjusted.

(b) Level the instrument so that, with the sector free, the line on the sector indicating the vertical from the point of suspension coincides with a corresponding point on the base of the instrument, usually placed on the stop mechanism. After leveling, operate the instrument several times with nothing in the jaws, the movable jaw being closed, to ascertain if the pointer registers zero with no load. If zero is not registered, the pointer stop should be suitably adjusted until the zero reading is obtained. If it is necessary to move the pointer stop, the pointer friction should be checked as follows: Set the pointer at the zero reading on the scale before releasing the sector and after release see that the pointer is not pushed more than three scale divisions beyond the zero. A reading of more than three divisions indicates excessive pointer friction and the pointer should be removed, the bearing wiped clean, and a trace of oil or petroleum jelly applied. When the pointer friction has been reduced, finally adjust the pointer stop.

#### Calibration of Apparatus

4. (a) Level and adjust the instrument on a level sheet of plate glass and clamp a known weight in grams,  $W$ , to the radial edge of the sector beneath the jaws. The center of gravity of the weight (including means of attaching) shall be previously marked by a punched dot on the face of the weight that is to be in the front of the instrument.

(b) Raise and set the sector as for tearing a sheet and, by means of a surface gage or other convenient means,

measure the height in centimeters,  $H$ , of the center of gravity of the weight above the glass plate. Then release the sector, allow it to swing, and note the pointer reading. Without touching the pointer, raise the sector until the edge of the pointer just meets with its stop, in which position again determine the height in centimeters,  $h$ , of the center of gravity of the weight above the glass plate.

(c) The work done is  $W(H - h)$  gram-centimeters. The pointer reading for the standard instrument and method specified should be as follows:

$$\frac{W(H - h)}{137.6}$$

(d) Five weights from 75 to 400 g. form a suitable range for the calibration, one or more being clamped on the edge of the sector in different positions, the work done in raising each being calculated and added together.

(e) A record shall be made of deviations of the indicated readings and corresponding corrections made in the test results.

(f) It is unnecessary to repeat the calibration of the instrument provided it is kept in adjustment and no parts become changed or worn, but the tearing distance, which equals 43 mm. (1.69 in.) shall be checked prior to each series of tests and adjusted if necessary.

#### Test Specimens

5. Test specimens shall be cut accurately in each principal direction of the paper about 76 mm. (3 in.) in length by exactly 63 mm. (2.5 in.) in width with the slit to be cut 20 mm. (0.8 in.) in length, leaving exactly 43 mm. (1.69 in.) between the end of the slit and the edge of the specimen.

#### Test Conditions

6. Samples shall be conditioned in

accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials<sup>3</sup> before the test specimens are cut. All test specimens shall be brought to a standard condition prior to testing in accordance with A.S.T.M. Method D 685, and all tests shall be made under the standard atmospheric conditions.

### Procedure

7. (a) Level and adjust the testing machine, if necessary, before each set of tests.

(b) Place the test specimen midway in the clamps with its upper edge parallel to the top of the jaws and so that the initial slit is at right angles to the top of the jaws (Note 2). Make alternate tests with the wire sides of the sheets comprising the test specimen facing opposite directions. Test enough sheets so that, when torn together (Note 3), the scale readings are between 20 to 60. Record the number of sheets so tested (1 to 16 sheets may be used). Make not less than five tests in each principal direction of the paper (Notes 4 and 5).

(c) If the mean value of the lowest and the highest reading differs from the average of all the readings by more than 10 per cent, test additional specimens until there is agreement within these limits. Discard an isolated very high or low result that is not repeated in duplicate when a consistent average has been obtained without the abnormal reading. Reject readings obtained

where the tear deviates more than 10 mm. ( $\frac{3}{8}$  in.) from the line of the initial slit. If results deviate more than this, a notation should be made and the deviation reported. If the side of the specimen above the movable clamp rubs against the sector as a tear is made, reject that reading.

### Calculation and Report

8. (a) Report the results as the force in grams required to tear a specimen. Since the scale readings are made one sixteenth of the actual values, the tearing resistance values shall be calculated by multiplying the average instrument reading (corrected if necessary for calibration error) by 16 and dividing by the number of sheets torn at one time. Report the results as follows:

Result	Report to Nearest
Below 10 units.....	0.1 g.
10 to 19.9.....	0.2 g.
20 to 49.9.....	0.5 g.
50 and above.....	1 g.

(b) Report the average, maximum, and minimum results for both principal directions of the paper and also the number of sheets torn at one time. Report results obtained on strips torn in the machine direction as resistance to tearing in the machine direction, and report results obtained on strips torn across the machine direction as resistance to tearing in the cross direction.

### Reproducibility of Results

9. Duplicate determinations on different sets of specimens from the same shipment and on different testing instruments should agree with each other within 7 per cent.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## EXPLANATORY NOTES

NOTE 1.—This method determines the average force in grams required to tear a sheet by measuring the work done in tearing a number of sheets together through a fixed distance. The scale indicates the ratio of the work done (in gram-centimeters) in tearing the specimen to the total tearing length of 16 sheets. This total tearing length is 16 (sheets) by 4.3 cm. (tearing length per sheet) by 2 = 137.6 cm., the factor 2 being included because in order to tear a sheet through a given distance, the tearing force has to be applied through twice that distance.

NOTE 2.—Perforating the specimens with a hole so that its edge is 4.3 cm. from their edges and cutting or tearing with the fingers the other side of specimens to the hole before inserting them in the instrument, gives the same results as slitting the specimen with the knife and is to be preferred for accurate work if a suitable punch having a jaw 4.3 cm. in depth from the inside edge of the plunger is available.

NOTE 3.—The work done in tearing a number of sheets of paper includes a certain amount of work to continuously bend the paper as it is torn, to provide for the rubbing of the torn edges of the specimen together, and to lift the sample of paper. Consequently, it is necessary to specify definite empirical requirements for both the apparatus and the method to keep the additional work not used for tearing to approximately a definite quantity. With jaws arranged so that after slitting the clamped edges are at right angles with one of the edges pointing away from the tearing direction the readings may be

very slightly increased, whereas if one of the edges is clamped at right angles so as to point towards the tearing direction, the readings may be decreased slightly (up to 5 per cent) from the standard. The influence of the number of specimens torn at one time and also their size often appreciably affects the total work done; consequently, for accurate work, the method should be followed as closely as possible.

NOTE 4.—As described by Carson and Snyder (Note 5), the capacity of the Thwing-Elmendorf tearing tester may be doubled by attaching to the pendulum in a suitable manner an auxiliary weight of 2501 g.

NOTE 5.—The following references may be of interest in connection with this method:

- A. Elmendorf, "Strength Test for Paper," *Paper*, Vol. 26, p. 302 (April 21, 1920).  
P. L. Houston, "A Supplementary Study of Commercial Instruments for Determining the Strength of Paper," *Paper Trade Journal*, Vol. 74, No. 10, p. 43 (March 9, 1922).  
Mallett and Marx, "The Calibration of the Elmendorf Tearing Tester," *Proceedings, Technical Section, Paper Makers' Assn. of Great Britain, Ireland* 4, p. 212 (March, 1924).  
F. T. Carson and L. W. Snyder, "Increasing the Capacity of the Elmendorf Tearing Tester," *Paper Trade Journal*, Vol. 86, No. 13, p. 57 (March 29, 1928).  
J. d'A. Clark, "Calibration of the Elmendorf Tearing Tester," *Technical Association Papers, Technical Assn. Pulp and Paper Industry, Series XV*, Vol. 1, p. 262 (1932).

## *Standard Method of Test for* BULKING THICKNESS OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 527 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 527; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test for bulking thickness covers the procedure for measuring the average thickness of paper when placed in a pile, as for use in books.

### Bulking Thickness

2. The bulking thickness of paper or paperboard is the average single-sheet thickness in thousandths of an inch when a pile of several sheets is placed under a steady pressure of 7 to 9 psi. between two circular and parallel plane surfaces, the smaller of which has an area of approximately 0.25 sq. in. (160 sq. mm.).

NOTE.—In general for ordinary papers, the average bulking thickness is approximately the same as the single-sheet thickness but usually from about 2 to 6 per cent lower. Also, depending on the type of paper, a variation in pressure of from 5 to 10 psi. causes a corresponding decrease in the results by about 3 per cent. Halving the area of the presser foot

while maintaining the unit pressure constant results in an increase of from 1 to 3 per cent.

### Apparatus

3. The micrometer shall conform to the following requirements:

(a) It shall have two plane faces, the smaller of which is circular and 0.25 to 0.33 sq. in. (160 to 215 sq. mm.) in area, corresponding to a diameter of 0.56 to 0.65 in. (14.3 to 16.5 mm.). The faces shall be parallel to within 0.0002 in. (0.005 mm.) and constrained to move apart in an axis perpendicular to themselves.

(b) When the specimen is clamped between the faces, it shall be under a steady pressure of 7.0 to 9.0 psi. (0.40 to 0.63 kg. per sq. cm.).

(c) The distance between the graduations on the dial shall be such as to permit of estimating the thickness to 0.0001 in. or less.

(d) The micrometer shall be such as to repeat its readings to within 0.0001 in. at zero setting or on a steel gage block.

(e) Measurements made on standard steel thickness gages shall be within the following permissible variations:

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

Intervals	Permissible Variation of Reading from Actual Thickness of Standard Steel Gage
0 to 0.25 mm. (0 to 0.01 in.) . . . .	0.0025 mm. (0.0001 in.)
Over 0.25 to 1.02 mm. (0.01 to 0.04 in.) . . . . .	0.0051 mm. (0.0002 in.)
Over 1.02 to 3.05 mm. (0.04 to 0.12 in.) . . . . .	0.0102 mm. (0.0004 in.)

### Calibration

4. (a) *Parallelism of Faces*.—A hard steel ball about  $\frac{1}{16}$  in. in diameter, fixed firmly in a thinner flat piece of metal to serve as a handle, shall be placed at different points on the anvil and the readings noted.

(b) *Accuracy of Readings*.—The micrometer shall be set to zero and standard gages shall be placed between the plane faces, and the corresponding dial readings observed over the range to be used. For precision measurements, standard steel gage blocks having an accuracy of 0.00001 in. shall be used. For bulking thickness measurements the commercial feeler gages may be used, but the accuracy of these shall be determined to the nearest 0.0001 in. by means of a screw micrometer of known accuracy.

(c) *Pressure Between Faces*.—The force required to just prevent the movement of the plunger foot, from a reading corresponding to about the average paper thickness tested to a lower reading, shall be determined with a suitable balance, and the contact pressure determined with this force. For example, where the presser foot projects through the top of the apparatus, a fine copper wire is attached to it and by means of a coarse balance or a calibrated spring, the force necessary to prevent the closing of the foot shall be measured.

Alternatively, a stirrup may be used, made of a flat metal plate having a hole larger than the diameter of the micrometer foot, covered at the bottom of the plate by a thin metal disk of about the average thickness of the paper to be measured. The stirrup shall be suspended from a suitable balance.

### Test Specimen<sup>3</sup>

5. The test specimen shall consist of not less than 10 sheets and shall have a minimum dimension, if possible, of not less than 2 in. Any necessary non-compliance with this requirement shall be noted in the test report as a minor deviation. The sheets shall be taken from the sample in such a way as to be thoroughly representative of it, and shall be free from creases. They shall have been conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685) of the American Society for Testing Materials.<sup>4</sup>

### Procedure<sup>3</sup>

6. (a) The test shall be made in an atmosphere conditioned in accordance with Tentative Method D 685.<sup>4</sup>

(b) A pack of paper sheets not less than 0.1 in. in thickness shall be tested in not less than ten different places evenly distributed over the pack. The pack shall be placed between the jaws of the micrometer and the presser foot lowered as gently as possible upon the surface of the paper with its edge at least  $\frac{1}{4}$  in. (0.6 mm.) from the edge of the paper.

### Report

7. The bulking thickness shall be reported in decimals of an inch, being the average thickness of the pack divided by the number of sheets in it, and shall be expressed to the nearest 0.00005 in. (0.001 mm.).

### Reproducibility of Results

8. Duplicate determinations from different sets of samples from the same shipment and on different instruments should agree within 3 per cent.

<sup>3</sup> Editorially revised in 1942.

<sup>4</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

## *Standard Methods of Test for* THICKNESS OF PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 645 - 43

ADOPTED, 1943.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 645; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover the procedures for determining the thickness of all kinds of paper and paper products except crepe paper and corrugated board. Five methods are covered, applicable to the following types of paper:

*Method A*, for the general run of papers and paper products.

*Method B*, for the general run of paper products. The use of the spring-actuated instrument is permissible as an alternative to the apparatus prescribed in method A, but is not recommended.

*Method C*, for soft, low density papers, such as roofing felt.

*Method D*, for electrical insulating papers.

*Method E*, for paper 2 mils (0.002 in.) and under in thickness.

### Thickness

2. Thickness as measured in these methods is defined as the thickness of a single sheet, in contradistinction to "bulking thickness," which is the thick-

ness of a pile of sheets determined in accordance with the Standard Method of Test for Bulking Thickness of Paper (A.S.T.M. Designation: D 527) of the American Society for Testing Materials.<sup>3</sup>

### Apparatus

3. The apparatus shall consist essentially of two plane parallel faces which can be moved apart or together along an axis perpendicular to themselves. In use, one of these faces (the anvil) shall be held stationary, the specimen shall be placed over it, and the other face (the presser foot) which is circular moved towards it until it exerts a predetermined pressure on the specimen. When this condition has been reached, the distance between the two faces shall be read on a suitable device and recorded as the thickness of the specimen.

(a) *Method A*.—The diameter of the presser foot shall be not less than 0.56 in. nor more than 0.65 in. The force shall be exerted by gravity acting on the presser foot and the moving parts connected therewith, and shall be such that the pressure is  $8 \pm 1$  psi.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1941 to 1943.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) *Method B.*—The diameter of the presser foot and the pressure shall be the same as for method A, but the force shall be exerted by a spring instead of by gravity.

(c) *Method C.*—The presser foot and actuating force shall be the same as that prescribed for method A but the pressure shall be  $4 \pm 1$  psi.

(d) *Method D.*—A machinist's rommicrometer conforming to the requirements for the instrument prescribed in method A of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

(e) *Method E.*—The apparatus prescribed for either method A or method D may be used.

#### Calibration

4. (a) *Parallelism of Faces.*<sup>3a</sup>—Place a hard steel ball about  $\frac{1}{8}$  in. in diameter, fixed firmly in a thinner, flat piece of metal to serve as a handle, between the presser foot and the anvil and note the readings at different points on the anvil.

(b) *Accuracy of Readings.*—Set the instrument to zero and place standard gage blocks having an accuracy of 0.00001 in. between the plane faces and observe the corresponding dial readings over the range to be used.

(c) *Pressure Between the Faces.*—Determine the force required to just prevent the movement of the presser foot from a reading approximately corresponding to the average thickness of the paper to be tested to a lower reading with a suitable balance and determine the contact pressure with this force. For example, attach a fine copper wire to the presser-foot where it projects through the top of the apparatus, and, by means of a coarse balance or a calibrated spring, measure the force necessary to prevent the closing of the foot. Alternatively, a

stirrup may be used made of a flat metal plate having a hole larger than the diameter of the micrometer foot, covered at the bottom of the plate by a thin metal disk of about the average thickness of the paper to be measured. Suspend the stirrup from a suitable balance.

(d) *Micrometer.*—Calibrate the micrometer used in methods D and E in accordance with the procedure described in Methods D 374.<sup>3</sup>

#### Sampling

5. The material shall be sampled in accordance with the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585) of the American Society for Testing Materials.<sup>3</sup>

#### Test Specimens<sup>4</sup>

6. Ten specimens shall be tested, each having a width of not less than 2 in. If it is not possible to secure specimens 2 in. in width, the width shall be stated in the report. Test specimens shall be taken from samples representative of the shipment and shall be free from creases.

#### Conditioning

7. Specimens shall be brought to equilibrium with an atmosphere of known temperature and humidity and shall be tested in that atmosphere.

#### Procedure

8. (a) *Methods A, B, and C.*—Place the specimen between the jaws of the measuring device and lower the presser-foot as gently as possible upon the surface of the paper, with its edge at least  $\frac{1}{4}$  in. from the edge of the paper. Determine the thickness of each of the ten specimens in each of two different places. If the mean between the maximum and minimum of the 20 results differs from the average of all 20 by more than plus

<sup>3a</sup> Editorially revised in January, 1947.

<sup>4</sup> See also Section 8 (c).



or minus 5 per cent, measure enough additional specimens to obtain agreement within these limits.

(b) *Method D.*—Determine the thickness of electrical insulating papers in accordance with the procedure described in method A of Methods D 374.<sup>3</sup>

(c) *Method E.*—Determine the thickness in accordance with the procedure described in either Paragraph (a) or (b). Determine the thickness of at least ten specimens of ten sheets each (Note).

NOTE.—For papers having a thickness of 2 mils or less a single specimen shall consist of a pile of ten sheets. If a micrometer is used to measure the thickness of a single sheet of this thinness, the error in reading the instrument is likely to make a significant difference in the

result. If a pile of ten sheets is measured and the result divided by ten, there will also be an error due to the "piling effect," but the latter error will, in general, be smaller than the former.

### Report

9. The report shall include the following:

- (1) Method used for determining thickness,
- (2) Relative humidity and temperature of conditioning atmosphere,
- (3) Number of specimens tested, if more or less than ten,
- (4) Width of the specimens, if less than 2 in., and
- (5) Maximum, minimum, and average thicknesses measured to the nearest 0.0001 in.

## *Standard Method of Test for*

### TIME OF PENETRATION BY WATER OF SIZED PAPER AND PAPER PRODUCTS (DRY INDICATOR METHOD)<sup>1</sup>



A.S.T.M. Designation: D 779 - 46

ADOPTED, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 779; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. This method of test covers the procedure for determining the time of penetration by water of sized paper and paper products. It consists of bringing one side of the test specimen in contact with water, and finding the time required for the water to pass through the specimen as indicated by the development of color in an indicator powder consisting of a mixture of water-soluble dye, sugar,<sup>3</sup> and starch (Note 1).

#### Apparatus

2. (a) Any form of apparatus may be used which conforms to the following requirements:

(1) One surface of the specimen shall be wetted uniformly at the moment the count of time is begun,

(2) An indicator on the opposite surface shall be continuously visible, and

(3) No moisture shall reach the indicator except that which passes through the specimen from the wetted surface, none of which shall escape contact with the indicator.

(b) The following apparatus has been found suitable for testing paper and paperboard (Note 2):

(4) *For Papers Having a High Degree of Water Resistance.*—A vessel of water of sufficient area for floating several specimens simultaneously, immersed in a water bath in which the temperature of the water in the vessel can be maintained at 22.5 to 23.5 C.; a sealing medium prepared by melting together and mixing thoroughly equal parts of wax and rosin; and a medicine dropper.

(2) *For Papers That Require a Short-Time Test (Note).*—The float arrangement shown in Fig. 1 is suitable for short-time tests. It shall be made of a thin-walled aluminum pan about

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1944 to 1946.

<sup>3</sup> The sugar serves the two-fold purpose of masking the color of the particles of dye as long as they are dry, and of absorbing the transuded moisture and holding it in close proximity to the dye. The starch increases the stability of the mixture.

5 in. in diameter and 1 in. in height, with a hole 2 in. in diameter cut in the bottom. The surface shall be coated with paraffin or wax to make it more water-repellant. The watch glass shall be clamped over the specimen by means of a wire-frame clamp hinged at one side and fastened under a spring at the opposite side. In the absence of the float, such papers may be supported by a hollow cylinder having the upper end barely submerged under the surface of the water in a suitable vessel, while the watch glass is placed over the indicator.

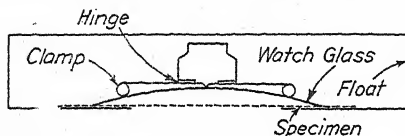


FIG. 1.—Cross-Section of Float.

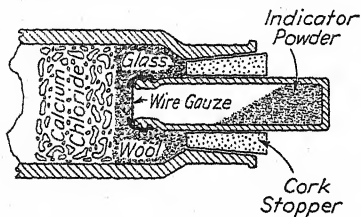


FIG. 2.—Longitudinal Section Showing Construction of Desiccator and Shaker.

Such a cylinder may also be found useful in supporting an occasional paper whose surface is so easily wetted that it would be difficult to float it unsupported when prepared as shown in Fig. 1.

NOTE.—Apparatus for a short-time test is needed when an edge seal is not required and when only one specimen can be tested at a time.

(3) *Shaker and Desiccator.*—A shaker and desiccator for the indicator (Fig. 2) prepared from a 10-ml. screw-top vial by cutting away most of the metal of the flat portion of the top, fitting a 70-mesh wire screen inside the top and screwing it back in place.

The desiccator shall be made of a small wide-mouthed bottle containing anhydrous calcium chloride covered with a layer of glass wool. A hole shall be bored in the cork stopper just large enough to admit the shaker. The bottle shall remain on its side and the vial inserted through the hole with the screened end inside. The assembly shall be kept in the usual type of laboratory desiccator when not in use.

(4) *Watch Glasses.*—Watch glasses 2-in. in diameter.

(5) *Stop Watch.*

### Reagents

3. (a) *Indicator.*—The water-transudation indicator shall be composed of pure cane sugar, pure soluble starch, and methyl violet dye.<sup>4</sup> Each ingredient shall be ground separately until it passes through a No. 100 sieve, and then dried completely in a desiccator over  $\text{CaCl}_2$  before making the mixture. When dry, the following proportions by weight shall be mixed:

Sugar.....	45
Soluble starch.....	5
Dye.....	1

The ingredients shall be mixed by screening repeatedly through a No. 60 sieve until the mixture is uniform. Mixing the components of the indicator by screening is essential to avoid discoloration, which occurs if they are ground together. The indicator shall be kept in a desiccator when it is not being used.

(b) *Water.*—Water of fair purity, such as the usual drinking water, is suitable.

### Test Specimens

4. (a) Test specimens shall be cut from different portions of the sample in

<sup>4</sup> DuPont N.E. methyl violet, National Aniline and Chemical Co. 2 B.P. methyl violet, or equivalent, is recommended for this purpose. Indicator mixture from M. Ames Chemical Works, Glens Falls, N. Y., or equivalent, is also recommended.

such a way as to be thoroughly representative of it. They shall be free from folds, wrinkles, or other blemishes not commonly inherent in the paper. A convenient size is about 2.5 in. square. The specimens shall be so prepared that five are tested from the wire side and five from the felt side. The expression "from the wire side" (or felt side) shall be interpreted to mean that water is applied to the side designated and the penetration proceeds therefrom.

(b) The test specimens shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>5</sup>

### Procedure

5. (a) Make the tests in an atmosphere conditioned in accordance with A.S.T.M. Method D 685 and with the temperature of the water maintained at 22.5 to 23.5 C.

(b) When testing materials having a high degree of water resistance place the test specimen on a level, smooth, surface, and sprinkle the indicator on the specimen by gently tapping the inverted shaker until a thin, even layer is formed, avoiding either a very sparse covering or one in which the powder is piled up (Note 3). Handle the test specimen with care once the powder is applied, for jarring or excessive tipping may cause rearrangement of the particles of powder, which then tend to form in clusters, thus destroying the intimate contact and regular thin layer that is desirable. As soon as the application of the layer of the indicator powder is completed, cover it with a 2-in. watch glass and keep it covered during the test. In testing material having a high degree of water resistance, use the apparatus described in Section 2 (b).

(c) For testing other forms of paper and paper products seal the edges of the specimen to prevent water from entering through the edges and reaching the indicator without going entirely through the material. Grasp the specimen at the center with thumb and forefinger as a pivot and dip into the melted wax-rosin mixture to a depth of  $\frac{1}{8}$  in., rotate 90 deg. and dip the next edge, and repeat until all four edges are saturated and sealed. (Homogeneous, nonlaminated materials will in general not require this edge seal.) When the wax has hardened, add the indicator

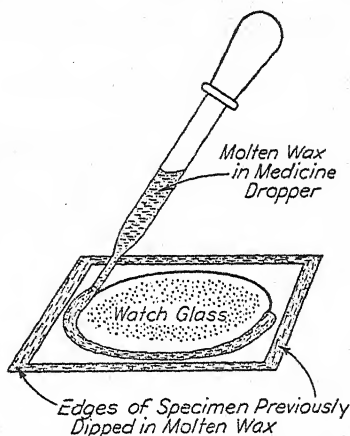


FIG. 3.—Watch Glass, Wax, and Indicator.

powder as described in Paragraph (b), with a 2-in. watch glass, and seal it with the melted wax-rosin mixture, applied with a medicine dropper, as illustrated in Fig. 3. Float the specimen thus prepared on a vessel of water, starting the stop watch at the instant of contact.

(d) For papers having a short testing period, when it is not necessary to seal the cover glass on, use the float shown in Fig. 1, or the submerged cylinder may be used to support the specimen and cover glass. In all cases, contact with the water shall be made at a slight angle to

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

avoid trapping air bubbles underneath the specimen. When testing materials having uneven surfaces, wet with water the surface to be in contact with water, using a soft brush, immediately before placing the specimen in the water. Examine each specimen after the test, and if there is evidence of uneven wetting, discard the result.

(e) *Measurement of Time of Transudation.*—Watch the test specimen continuously for a change in the color of the indicator, and measure the time interval from the instant of contact with the water until the rate of change in the color of the indicator is at a maximum. For most ordinary papers, this time interval is conveniently determined as the mean of the values corresponding to the development of pronounced color in one fourth and in three fourths of the area covered by the indicator. For materials on which the color develops uniformly over the whole area, it may be necessary to tabulate the values of the time interval against the intensity of the color to determine the maximum rate of change. In judging the end point on specimens requiring considerable time to reach the end point, note the following three general color phases: As it is first sprinkled on the test area the powder often shows a slight tinge of color other than the predominant white. This first color may appear slightly different on surfaces of different color; after the test has been in progress for such a time that the end point is reached, a more intense color is present which is a somewhat reddish

purple. Finally, if the specimen is left for a longer time than the proper end point, the reddish cast tends to disappear and the powder appears a dark purple, also, the particles often appear as if quite moist. Sometimes, when the specimens are unevenly resistant, there may be indications of two or possibly all three phases at once, but it should be easily ascertainable which is predominant. In any case, taking the first indication of change from the white should be avoided, allow enough change to take place to be positive. On the other hand, do not wait for the last purple phase, for once this is attained there is little further change to note with the passage of time. Artificial light, when used for viewing the test specimen, should be reasonably brilliant, but completely shaded from the eyes. Apply the light to one side of the specimen (thus avoiding reflection from the cover glass), and the eye view from the side next to the illuminant, to avoid interference of shadows cast by the indicator particles with the judgment of the color.

### Report

6. The report shall give the average, minimum, and maximum values of the time of transudation in minutes from the wire side and from the felt side.

### Reproducibility

7. Average test results by experienced operators should agree within 10 to 15 per cent.

## EXPLANATORY NOTES

NOTE 1.—This method is sometimes not applicable to materials containing large amounts of water-soluble components.

NOTE 2.—*Penoscope.*—This device can be used for the dry-indicator test. It consists of a cast brass chamber which is filled with water;

a hollow screw cap, with an opening for observation of the test specimen, in which the test specimen is inserted and then screwed tight against the chamber; and pipe connections at top and bottom of the chamber by means of which the water can be introduced, maintained

at a desired level, and removed. The suitability of this device for a given purpose should be determined by comparing the results obtained with it with those obtained by the standard method.

NOTE 3.—In the former case, the color change produced by too few dye particles may be insufficient to attract attention, unless they are exposed for a longer time than the proper end point. When the powder is placed too thickly, the upper layer may obscure changes in the portion in contact with the paper. Also, the greater total quantity of powder may require more time to change, since it would necessitate more moisture to affect all the dye.

NOTE 4.—Investigations of the use of higher temperatures in the dry-indicator test have shown that in general there is good correlation between transudation time and temperature. For products that show such relation, this is of value in shortening the test period, and in some instances in making the end point more distinct.

NOTE 5.—For further information on this method the following references should be consulted:

A. Abrams, *Paper Trade Journal*, Vol. 84, No. 3, January 30, 1927, pp. 44-47; *TS* pp. 38-41.

A. Abrams, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Vol. 12, No. 49 (1929); *Paper Trade Journal*, Vol. 88, No. 9, February 28, 1929, pp. 40-42; *TS* pp. 147-149.

F. T. Carson, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Vol. 8, p. 91 (1925); *Paper Trade Journal*, Vol. 80, No. 10, March 5, 1925, pp. 59-62; *TS* pp. 85-88.

F. T. Carson, *Technologic Paper No. 326*, Nat. Bureau Standards, September, 1926.

F. T. Carson and F. V. Worthington, *Paper Trade Journal*, Vol. 95, No. 16, October 20, 1932, pp. 34-36; *TS* pp. 188-190.

F. T. Carson, *Paper Trade Journal*, Vol. 98, No. 21, May 24, 1934, pp. 36-38; *TS* pp. 264-266.

P. W. Codwise, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Vol. 14, p. 175 (1931); *Paper Trade Journal*, Vol. 92, No. 10, March 5, 1931, pp. 55-57, *TS* pp. 138-140.

P. W. Codwise, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Vol. 15, p. 234 (1932); *Paper Trade Journal*, Vol. 94, No. 5, February 4, 1932, pp. 42-45; *TS* pp. 46-49.

P. W. Codwise, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Vol. 17, pp. 143-145 (1934); *Paper Trade Journal*, Vol. 98, No. 10, March 8, 1934, pp. 43-45; *TS* pp. 123-125.

P. W. Codwise, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Vol. 26, p. 165 (1943); *Paper Trade Journal*, Vol. 116, No. 9, March 4, 1943, pp. 30-33; *TS* pp. 90-93.

## Standard Method of Test for WATER-SOLUBLE ACIDITY OR ALKALINITY OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 548 - 41

ADOPTED, 1941.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 548; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. This method of test covers the procedure for determining the water-soluble acidity or alkalinity of paper.<sup>3</sup> The method is not applicable to highly-alkaline papers such as those containing casein or calcium carbonate, nor is it applicable to electrical insulating papers.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Grinder*.—A Koerner<sup>4</sup> or equivalent type grinder that will completely disintegrate the paper without heating or contaminating it.

(b) *Bath*.—A steam or oil bath which can be maintained at 100 C.

(c) *Balance*.—A balance sensitive to 1 mg.

(d) *Glassware*.—Acid- and alkali-resistant glassware.

### Reagents

3. (a) *Sodium Hydroxide* (0.01 N).—Dilute 100 ml. of 0.1 N NaOH to 1 liter with freshly boiled and cooled distilled water.

(b) *Hydrochloric or Sulfuric Acid* (0.01 N).

(c) *Phenolphthalein Indicator*.—Dissolve 1 g. of phenolphthalein in 100 ml. of 50 per cent alcohol; add 0.01 N alkali cautiously until a faint pink color appears; then just remove the color with a drop or two of 0.01 N acid.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, this method was published as tentative from 1939 to 1941.

<sup>3</sup> This method is based on the method for acidity described by S. Kohler and G. Hall in *The Paper Industry*, 7, No. 7; (October, 1925), with some modifications developed at the National Bureau of Standards. In this revised method, one extraction is specified instead of the three extractions originally specified, because Kohler subsequently found that one extraction is sufficient for classification of paper. See "Investigation into the Determination of Acidity and Copper Number in Paper," Meddelande 56 Statens Provingsanstalt, Stockholm, (1932). In this article, it is stated that the acid number thus obtained is about three-fourths of that obtained by three extractions.

Studies of this method and other acidity methods are reported by B. L. Wehmhoff and by P. F. Wehmer in *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, May, 1930, and May, 1931.

<sup>4</sup> The Koerner type of grinder is described in a paper by J. O. Burton and R. H. Rasch on "The Determination of the Alpha-Cellulose Content and Copper Number of Paper," *Journal of Research*, Nat. Bureau Standards, Vol. 6, No. 4, p. 603 (1931) (*Research Paper 295*).

### Test Specimen

4. The test specimen shall be cut from the sample in such a way as to be thoroughly representative. The specimen shall be completely disintegrated in the grinder and shall be of sufficient size to yield at least 15 g. of disintegrated material.

### Procedure

5. (a) Allow the ground test specimen to reach moisture equilibrium with the atmosphere of the balance case, and then weigh at the same time two 5-g. portions (to nearest 1 mg.) for the acid extraction and the moisture determination.

(b) Determine the moisture content on one portion in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (A.S.T.M. Designation: D 644) of the American Society for Testing Materials.<sup>5</sup>

(c) Transfer the other weighed portion to a 500-ml. Erlenmeyer flask and add 250 ml. of boiling water. In some cases, the fibers absorb water slowly and tend to float on the surface of the water. This may be avoided by first adding small portions of the water and shaking well until the fibers are thoroughly saturated. After the water is added, affix to the flask a stopper containing a narrow glass tube about 30 in. in length, which serves as a condenser. A soil-digestion flask which has a ground-glass stopper and condensing tube in one piece, or a rubber stopper covered with metal foil, may be used. Place the flask in a heating bath which will maintain the contents of the flask at 98 to 100 C. Heat at this temperature for 1 hr. while shaking occasionally. At the end of this period pour the contents of the flask into a Büchner funnel (without other filtering medium) and wash the fibers remaining

in the flask into the Büchner funnel with 10 ml. of water. Apply strong suction to the fibers, then cool the extract rapidly and titrate it as soon as it reaches room temperature. Add phenolphthalein indicator and, if the extract remains colorless, determine the acidity by titrating with 0.01 *N* NaOH until the first appearance of a permanent pink coloration. If on addition of phenolphthalein the extract has a pink color, determine the alkalinity by titrating with 0.01 *N* HCl or H<sub>2</sub>SO<sub>4</sub> until the color is just discharged. Make a blank titration on 250 ml. of the water heated for 1 hr. in the same bath and with the same glassware used for the extractions.

(d) Each test result used in the calculations shall be the average of not less than two determinations. Results of duplicate determinations shall agree within 0.01 per cent.

### Calculations and Report

6. Total acidity or alkalinity shall be expressed as a percentage of the moisture-free paper in terms, respectively, of sulfur trioxide (SO<sub>3</sub>), or in terms of sodium oxide (Na<sub>2</sub>O), to the nearest 0.01 per cent calculated as follows:

$$\text{SO}_3, \text{ per cent} = \frac{(T_1 - t) \times N_1 \times 0.04}{W} \times 100$$

Na<sub>2</sub>O, per cent

$$= \frac{(T_2 \times N_2 + t \times N_1) \times 0.03}{W} \times 100$$

where:

$T_1$  = milliliters of NaOH required to neutralize the extract,

$T_2$  = milliliters of acid required to neutralize the extract,

$t$  = milliliters of NaOH required to neutralize the blank,

$N_1$  = normality of NaOH solution,

$N_2$  = normality of acid solution, and

$W$  = weight of test specimen, less moisture.

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



## *Standard Methods of Test for* WIRE AND FELT SIDES OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 725 - 45

ADOPTED, 1945.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation D 725; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. (a) These methods of test cover four procedures for determining the wire and felt sides of paper. It is not always possible to make the distinction, this being particularly true of coated and other surface-treated papers, and of specialties made with variations in the usual paper-machine practice. When one of the procedures described in this method does not give a definite differentiation, it is advisable to try one or more of the other procedures.

(b) The wire side of paper is the side that was in contact with the wire of the paper machine; the other side is termed the felt side. It is desirable to have means of distinguishing between the two sides, because they generally differ in some physical characteristics.

### Procedure A

2. Fold over a sheet of the paper and observe the relative smoothness of the two sides. Often the pattern of diamond-shaped impressions of the machine wire can be seen, thus identifying

the wire side. In viewing the paper, hold it in a horizontal position with the light striking it at an angle of about 45 deg. and with the line of vision of the eye also at an angle of 45 deg. to the surface of the paper. Observation with a microscope is helpful (Note).

NOTE.—Making a black smudge or mark on the paper surface by wiping with a piece of typewriter carbon paper blackens the high spots on the paper surface and sometimes makes the wire marks more easily visible. To do this, hold a small piece of carbon paper between the thumb and middle or forefinger, so that the black surface of the carbon paper is pressed into the surface of the paper under test by whichever finger is used. Using considerable pressure, slowly pull the carbon paper along the surface of the paper, thus producing a black mark about  $\frac{1}{2}$  in. in width and 2 or 3 in. in length. During this operation, support the paper on a smooth surface, such as plate glass. Make such a mark on both sides of the paper across both the machine and cross directions as determined by the Standard Method of Test for Machine Direction of Paper (A.S.T.M. Designation: D 528).<sup>3</sup> Use a fresh spot on the carbon paper in making each mark.

### Procedure B

3. Dip the paper in water or in a weak sodium hydroxide solution, drain off the excess liquid, allow to stand for a

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Prior to adoption as standard, these methods were published as tentative from 1943 to 1945.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

few minutes, and again examine the two sides. This treatment tends to restore to paper the texture it had on the machine wire. View as described in Procedure A (Section 2). (If paper is dry, see Note, Section 2).

#### Procedure C

4. Hold a sheet of paper in one hand in such a way that the grain (machine) direction is parallel to the line of vision and the sheet surface is approximately horizontal. Holding the sheet in this position with one hand, pull upward with the other to start a tear in the sheet so that the line of tear follows the grain of the paper. As the tear is being made, gradually guide it so that it moves in the cross direction and toward the outer edge of the sheet, producing a tear line following a curved path. Turn the sheet over so that the opposite side is uppermost, and make a similar tear in the sheet. Observe the feathering caused by the splitting of the sheet at the edge of the two tears which have been made. One of these tears will show a distinct feather edge as compared to the other, especially in the curved portion, where the line of tear departs from the grain or machine direc-

tion and moves toward the cross direction. The tear with the more feathered edge is always made when the wire side of the sheet faces upward.

NOTE.—Procedure C is very useful as it often gives a positive determination when the other simpler procedures fail. The successful use of it, however, requires a certain amount of experience and it is recommended that such experience be gained by applying it to papers of known wire side and felt side.

#### Procedure D

5. Cut specimens approximately 1 in. in width and 2 in. in length, with the long dimension at right angles to the machine direction of the paper. Place the specimens in a drying oven at approximately 100 C., or in a desiccator over a suitable drying agent, such as anhydrous calcium chloride. It is essential that both sides be exposed alike to the drying air, and they shall be suspended or otherwise held so that they can curl without restraint. The direction of curl under these conditions will furnish a reliable means of distinguishing between the two sides of most papers. On drying, any pronounced curl of the specimen will be toward the wire side, with the axis of the curl parallel to the machine direction of the paper.

## *Standard Specifications for* A.S.T.M. THERMOMETERS<sup>1</sup>



A.S.T.M. Designation: E 1 - 46

ADOPTED, 1939; REVISED, 1942, 1944, 1946.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation E 1; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These specifications cover glass thermometers graduated in Centigrade or Fahrenheit degrees and frequently specified in methods of the American Society for Testing Materials. The various thermometers covered are listed in Table I.

NOTE.—The Tentative Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 - 46 T)<sup>3</sup> cover other thermometers that are intended, when adopted, to be incorporated in this standard.

### Requirements

2. The individual thermometers shall conform to the detailed specifications given in Table I and to the general requirements specified in Sections 3 to 11.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> These specifications represent a consolidation of the requirements for A.S.T.M. thermometers which were previously published in individual methods of test issued by the American Society for Testing Materials.

<sup>3</sup> See p. 1281.

NOTE.—For the purpose of interpreting these specifications the following descriptions of terms apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

The top of the thermometer is the top of the finished instrument.

### Type

3. Each thermometer shall be of the etched glass stem type unless otherwise specified in Table I.

### Stem

4. The stem shall be made of suitable thermometer tubing and shall have a plain front and enamel back.

### Bulb

5. The bulb shall be made of Corning normal or equally suitable thermometric glass.

### Graduations

6. All graduation lines, figures, and

letters shall be clear-cut and distinct. On partial immersion thermometers a line of approximately the same length as the longest graduation line shall be etched on the front of the thermometer at the distance above the bottom of the bulb as specified in Table I.

### Special Marking

7. The special markings on the thermometers in the form of an inscription as specified in Table I shall be in capital letters without the use of periods. In addition to the special markings prescribed in Table I, each thermometer shall be marked with a serial number and the manufacturer's name or trade mark etched on the stem.

### Scale Error

8. The error at any point of the scale up to the temperature limit prescribed in Table I when the thermometer is standardized in accordance with Section

9 shall not exceed the values prescribed in Table I.

### Standardization

9. The thermometer shall be standardized immersed in the testing bath to the top of the mercury column unless other conditions of immersion are prescribed in Table I, and at the temperatures prescribed in Table I. (See Explanatory Notes)

### Test for Permanency of Range

10. The test for permanency of range shall be made at the temperature prescribed in Table I and under the immersion conditions specified for the thermometer. The accuracy after this test shall be within the limit specified.

### Case

11. The thermometer shall be supplied in a suitable case on which shall appear the marking prescribed in Table I.

## EXPLANATORY NOTE ON PERIODIC CHECKING ON CHANGES OF BULB VOLUME OF GLASS THERMOMETERS

A mercury in glass thermometer, if properly made and used, is an extremely reliable instrument but small changes in volume of the thermometer bulb may occur, even though the thermometer may have been carefully annealed or aged.

Usually the bulb contracts slowly and as a result the thermometer may read higher after it has been placed in service than it did originally. These changes are more appreciable for short-range thermometers graduated in fractional degrees, and the users of such thermometers should check such instruments from time to time. When first received the thermometers should be checked every week or so, later these time intervals may be lengthened if the changes are not of consequence.

The ice-point method (reading in melting ice at 0 C. or 32 F.) has been found to be the most convenient and at the same time the most accurate method for checking changes in bulb volume. Other temperatures, such as the boiling point of water and of other pure substances, have been proposed for "fixed points," but have

not been found entirely satisfactory owing to the difficulties of accurately obtaining these fixed temperatures.

Short-range thermometers graduated in fractional degrees may or may not be provided with an ice point reading if this temperature is not included in the given range. This extra point can be incorporated in the thermometer, but may be omitted for the sake of economy. In the case of distillation thermometers intended to be inserted in the neck of a flask, the ice-point graduation may be undesirable since the contraction chamber between the ice point and the first graduation may contain enough mercury to cause uncertain and variable readings. For this reason the ice-point graduation, unless it is an integral part of the range, is generally omitted from the specifications for distillation thermometers.

In the absence of the ice point other means must be provided for detecting changes in the readings, especially if the thermometer is fractionally graduated. The method most commonly used involves the comparison at one or

more temperatures in a well-stirred, properly constructed liquid bath of the thermometer to be checked against another standardized thermometer of similar specifications to the test thermometer but provided with an ice point.

Where several thermometers make up a series or set for some specific method and these thermometers do not have ice points, a similar set may be provided with ice points, and these

should be kept for use as reference standards for checking routine test thermometers.

Detailed instructions for making such comparisons and specifications for suitable testing apparatus, as well as other data on this subject, will be found in the paper by E. F. Mueller and R. M. Wilhelm, "Methods of Testing Thermometers," *Proceedings, Am. Soc. Testing Mats.*, Vol. 38, Part I, p. 493 (1938).

#### EXPLANATORY NOTE ON ICE POINT DETERMINATIONS OF GLASS THERMOMETERS

Two methods have been in use for determining the ice point on thermometers graduated fractionally and with an open scale, such as those of the Kinematic type. One method requires that the thermometer remain at approximately room temperature for 72 hr. before the ice point is taken. The other stipulates that the ice point reading be taken immediately or within a definitely stated short period after heating to the test temperature. Changes in the correction of the thermometer may be observed by determining the changes in the ice point by either method.

Since it may not be practical to wait until the thermometer has rested for three or more days,

the method as outlined below is suggested. These instructions are given in the form of a note which should appear in the table of corrections for the thermometer.

NOTE.—The tabulated corrections apply for the condition of immersion indicated provided the ice point reading taken after heating to.....for not less than three minutes is.....

If the ice point reading (taken in not less than 2 min. and not more than 5 min. after removal of the thermometer from the heated bath) is found to be higher (or lower) than stated, all other readings will be higher (or lower) to the same extent.

(See Table I, pp. 457 to 470.)

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS.

Name.....	A.S.T.M. Partial Immersion <sup>a</sup>		A.S.T.M. Partial Immersion <sup>b</sup>	
A.S.T.M. Thermometer...	1C - 39	1F - 39	2C - 39	2F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-20 to +150 C.	0 to +300 F.	-5 to +300 C.	+20 to +580 F.
Subdivisions.....	1 C.	2 F.	1 C.	2 F.
Total Length....	303 to 307 mm.		379 to 383 mm.	
Stem Diameter....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	5.0 to 6.0 mm.		5.0 to 6.0 mm.	
Bulb Length.....	19 to 25 mm.		10 to 15 mm.	
Bottom of Bulb to Graduation Line at..... Distance.....	-18 C. 90 to 100 mm.	0 F.	0 C. 100 to 110 mm.	32 F.
Top of Thermometer to Graduation Line at..... Distance.....	150 C. 20 to 35 mm.	300 F.	300 C. 25 to 50 mm.	572 F.
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	5 C.	10 F.	5 C.	10 F.
Graduations Numbered at Each Multiple of.....	10 C.	20 F.	10 C.	20 F.
Immersion (See Section 6)	76 mm.	3 in.	76 mm.	3 in.
Special Marking on Thermometer (See Section 7).....	ASTM 76-MM IMM	ASTM 3-IN IMM	ASTM 76-MM IMM	ASTM 3-IN IMM
Scale Error at any point when standardized shall not exceed.....	0.5 C.	1 F.	1 C.	2 F.
Standardization.....	c	c	c	c
Marking on Case.....	A.S.T.M. -20 to +150 C., 76-mm. immersion	A.S.T.M. 0 to +300 F., 3-in. immersion	A.S.T.M. -5 to +300 C., 76-mm. immersion	A.S.T.M. 20 to 580 F., 3-in. immersion

<sup>a</sup> These requirements were previously specified in A.S.T.M. Standard D 182.<sup>b</sup> These requirements were previously specified in A.S.T.M. Standard D 183.<sup>c</sup> The thermometer shall be standardized for 76-mm. or 3-in. immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
50 C.....	35 C.	122 F.....	94 F.
100 C.....	48 C.	212 F.....	118 F.
150 C.....	55 C.	302 F.....	131 F.

<sup>d</sup> The thermometer shall be standardized for 76-mm. or 3-in. immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
50 C.....	35 C.	122 F.....	94 F.
100 C.....	49 C.	212 F.....	120 F.
150 C.....	61 C.	302 F.....	142 F.
200 C.....	70 C.	392 F.....	158 F.
250 C.....	76 C.	482 F.....	169 F.
300 C.....	80 C.	572 F.....	176 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Partial Immersion <sup>a</sup>		A.S.T.M. Acid Heat <sup>b</sup>	
A.S.T.M. Thermometer...	3C - 39	3F - 39	4C - 39	4F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-5 to +400 C.	+20 to 760 F.	0 to 105 C.	30 to 220 F.
Subdivisions.....	1 C.	2 F.	0.5 C.	1 F.
Total Length.....	404 to 408 mm.		378 to 384 mm.	
Stem Diameter.....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	5.0 to 6.0 mm.		5.0 to 6.0 mm.	
Bulb Length.....	10 to 15 mm.		10 to 15 mm.	
Bottom of Bulb to Graduation Line at..... Distance.....	0 C. 100 to 110 mm.	32 F.	0 C. 200 to 215 mm.	32 F.
Top of Thermometer to Graduation Line at..... Distance.....	400 C. 25 to 50 mm.	752 F.	105 C. 25 to 40 mm.	220 F.
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	5 C.	10 F.	1 C.	5 F.
Graduations Numbered at Each Multiple of.....	10 C.	20 F.	5 C.	10 F.
Immersion (See Section 6)	76 mm.	3 in.	152 mm.	6 in.
Special Marking on Thermometer (See Section 7).....	ASTM 76-MM IMM	ASTM 3-IN IMM	ASTM ACID HEAT 152-MM IMM	ASTM ACID HEAT 6-IN IMM
Scale Error at any point when standardized shall not exceed.....	{ 1 C. up to 301 C. 1.5 C. over 301 C.	{ 2 F. up to 574 F. 3 F. over 574 F.	0.5 C.	1 F.
Standardization.....	c	e	d	d
Test for Permanency of Range (See Section 10)	subject to 10 C. below max. scale temp. for 24 hr.	subject to 20 F. below max. scale temp. for 24 hr.	....	....
Marking on Case.....	A.S.T.M. -5 to +400 C., 76-mm. immersion	A.S.T.M. 20 to 760 F., 3-in. immersion	A.S.T.M. Acid Heat, 0 to 105 C.	A.S.T.M. Acid Heat, 30 to 220 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Standard D 184.<sup>b</sup> These requirements were previously specified in A.S.T.M. Method D 481.<sup>c</sup> The thermometer shall be standardized for 76-mm. or 3-in. immersion and for the following temperatures of the emergent mercury column. These stem temperatures have been chosen as corresponding, on the average, to those likely to occur in the use of the thermometer.

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C.....	50 C.	212 F.....	122 F.
200 C.....	75 C.	392 F.....	167 F.
300 C.....	89 C.	572 F.....	192 F.
400 C.....	94 C.	752 F.....	201 F.

<sup>d</sup> The thermometer shall be standardized at the ice point and at intervals of approximately 30 C. or 50 F. for 152 mm or 6-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
20 C.....	20 C.	70 F.....	70 F.
40 C.....	31 C.	100 F.....	86 F.
70 C.....	40 C.	150 F.....	104 F.
100 C.....	48 C.	212 F.....	118 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Cloud and Pour <sup>a</sup>		A.S.T.M. Low Cloud and Pour <sup>a</sup>	
A.S.T.M. Thermometer...	5C - 39	5F - 39	6C - 39	6F - 39
Liquid.....	mercury	mercury	toluene <sup>b</sup>	toluene <sup>b</sup>
Filling above Liquid.....	nitrogen gas	nitrogen gas	gas under pressure	gas under pressure
Temperature Range.....	-38 to +50 C.	-36 to +120 F.	-60 to +20 C.	-70 to +70 F.
Subdivisions.....	1 C.	2 F.	1 C.	2 F.
Total Length.....	220 to 224 mm.		227 to 231 mm.	
Stem Diameter.....	7.0 to 8.0 mm.		7.0 to 8.0 mm.	
Bulb Diameter.....	not greater than stem		5.0 to 6.5 mm.	
Bulb Length.....	not over 9.5 mm.		8.0 to 9.5 mm.	
Bottom of Bulb to Graduation Line at.....	-38 C.	-36 F.	-57 C.	-70 F.
Distance.....	120 to 130 mm.		120 to 130 mm.	
Top of Thermometer to Graduation Line at.....	+49 C.	+120 F.	+20 C.	+68 F.
Distance.....	19 to 25 mm.		35 to 45 mm.	
Expansion Chamber shall permit heating to.....	+100 C.	+212 F.	+60 C.	+140 F.
Top Finish.....	plain	plain	plain	plain
Longer Graduation Lines at Each.....	5 C.	10 F.	5 C.	10 F.
Graduations Numbered at Each Multiple of.....	10 C.	20 F.	10 C.	20 F.
Immersion (See Section 6)	108 mm.	4½ in.	76 mm.	3 in.
Special Marking on Thermometer (See Section 7).....	108-MM IMM ASTM CLOUD AND POUR	4½-IN IMM ASTM CLOUD AND POUR	76-MM IMM ASTM LOW CLOUD AND POUR	3-IN IMM ASTM LOW CLOUD AND POUR
Scale Error at any point when standardized shall not exceed.....	0.5 C.	1 F.	1 C.	2 F.
Standardization.....	ice point, every 20 C.; and avg. temp. 21 C. of emergent stem	ice point, every 40 F.; and avg. temp. 70 F. of emergent stem	ice point, every 20 C.; and avg. temp. 21 C. of emergent stem	ice point, every 35 F.; and avg. temp. 70 F. of emergent stem
Marking on Case.....	A.S.T.M. Cloud and Pour, -38 to +50 C.	A.S.T.M. Cloud and Pour, -36 to +120 F.	A.S.T.M. Low Cloud and Pour, -60 to +20 C.	A.S.T.M. Low Cloud and Pour, -70 to +70 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Method D 97.<sup>b</sup> Toluene or other suitable liquid colored red with a permanent dye shall be used.



TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Low Distillation <sup>a</sup>		A.S.T.M. High Distillation <sup>b</sup>	
A.S.T.M. Thermometer...	7C - 39	7F - 39	8C - 42	8F - 42
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	0 to 300 C.	30 to 580 F.	0 to 400 C. <sup>c</sup>	30 to 760 F. <sup>c</sup>
Subdivisions.....	1 C. <sup>d</sup>	2 F.	1 C. <sup>d</sup>	2 F.
Total Length.....	378 to 384 mm.		378 to 384 mm.	
Stem Diameter.....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	5.0 to 6.0 mm.		5.0 to 6.0 mm.	
Bulb Length.....	10 to 15 mm.		10 to 15 mm.	
Bottom of Bulb to Graduation Line at.....	0 C.	32 F.	0 C.	32 F.
Distance.....	100 to 110 mm.		25 to 45 mm.	
Top of Thermometer to Graduation Line at.....	300 C.	572 F.	400 C.	752 F.
Distance.....	30 to 45 mm.		30 to 45 mm.	
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	5 C.	10 F.	5 C.	10 F.
Graduations Numbered at Each Multiple of.....	10 C.	20 F.	10 C.	20 F.
Immersion.....	total	total	total	total
Special Marking on Thermometer (See Section 7).	ASTM LOW DISTILLATION	ASTM LOW DISTILLATION	ASTM HIGH DISTILLATION	ASTM HIGH DISTILLATION
Scale Error at any point up to.....	300 C.	572 F.	370 C.	700 F.
when standardized, shall not exceed....	0.5 C.	1 F.	1 C.	2 F.
Standardization.....	ice point, every 50 C., and at 300 C.	ice point, every 100 F., and at 572 F.	ice point, every 50 C., and at 370 C.	ice point, every 100 F., and at 700 F.
Test for Permanency of Range (See Section 10).	subject to 280 to 290 C. for 24 hr.	subject to 540 to 560 F. for 24 hr.	subject to 360 to 370 C. for 24 hr.	subject to 680 to 700 F. for 24 hr.
Marking on Case.....	A.S.T.M. Low Distillation, 0 to 300 C.	A.S.T.M. Low Distillation, 30 to 580 F.	A.S.T.M. High Distillation, 0 to 400 C.	A.S.T.M. High Distillation, 30 to 760 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Methods D 86, D 216, and D 285.

<sup>b</sup> These requirements were previously specified in A.S.T.M. Methods D 20, D 86, D 246, D 370, and D 402, except for the revision adopted in 1942.

<sup>c</sup> Under certain test conditions, the bulb of the thermometer may be 28 C. (50 F.) above the temperature indicated by the thermometer, and at an indicated temperature of 371 C. (700 F.) the temperature of the bulb is approaching a critical range in the glass. It is therefore not desirable to use this thermometer under such conditions at indicated temperatures above 371 C. (700 F.) without checking the ice point.

<sup>d</sup> For purposes of calibration and certification two or three division markings shall be placed below the zero on the Centigrade scale thermometer.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Pensky-Martens, Low Range <sup>a</sup> A.S.T.M. Tag Closed Tester <sup>a</sup>		A.S.T.M. Pensky-Martens, High Range <sup>b</sup>	
	A.S.T.M. Thermometer...	A.S.T.M. Tag Closed Tester...	A.S.T.M. Thermometer...	A.S.T.M. Tag Closed Tester...
A.S.T.M. Thermometer...	9C - 39	9F - 39	10C - 39	10F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-7 to +110 C.	+20 to +230 F.	90 to 370 C.	200 to 700 F.
Subdivisions.....	0.5 C.	1 F.	2 C.	5 F.
Total Length.....	273 to 277 mm.		273 to 277 mm.	
Stem Diameter.....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	not greater than stem		not greater than stem	
Bulb Length.....	9.0 to 13 mm.		not over 10 mm.	
Bottom of Bulb to Graduation Line at..... Distance.....	-7 C. 75 to 90 mm.	+20 F.	93 C. 75 to 90 mm.	200 F.
Top of Thermometer to Graduation Line at..... Distance.....	+110 C. 25 to 40 mm.	+230 F.	371 C. 25 to 40 mm.	700 F.
Expansion Chamber.....		c	....	....
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	1 C.	5 F.	10 C.	25 F.
Graduations Numbered at Each Multiple of.....	5 C.	10 F.	20 C.	50 F.
Immersion (See Section 6)	57 mm.	2¼ in.	57 mm.	2¼ in.
Special Marking on Thermometer (See Section 7)	57-MM. IMM ASTM PM AND TAG	2¼-IN IMM ASTM PM AND TAG	57-MM IMM ASTM PM HIGH	2¼-IN IMM ASTM PM HIGH
Scale Error at any point when standardized shall not exceed.....	0.5 C.	1 F.	1 C.	2.5 F.
Standardization.....	d	d	e	e
Test for Permanency of Range (See Section 10)	....	....	subject to 360 to 370 C., for 24 hr.	subject to 680 to 700 F., for 24 hr.
Marking on Case.....	A.S.T.M. P.M. and Tag, -7 to +110 C.	A.S.T.M. P.M. and Tag, +20 to +230 F.	A.S.T.M. P.M. High, 90 to 370 C.	A.S.T.M. P.M. High, 200 to 700 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Methods D 56 and D 93.<sup>b</sup> These requirements were previously specified in A.S.T.M. Method D 93.<sup>c</sup> The expansion chamber shall permit heating the thermometer at least 50 C. or 90 F. above the highest temperature on the scale.<sup>d</sup> The thermometer shall be standardized at the ice point and at intervals of approximately 30 C. or 50 F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
20 C.....	20 C.	70 F.....	70 F.
40 C.....	31 C.	100 F.....	86 F.
70 C.....	40 C.	150 F.....	104 F.
100 C.....	48 C.	212 F.....	118 F.

<sup>e</sup> The thermometer shall be standardized at intervals of approximately 50 C. or 100 F. for 57-mm. or 2¼-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C.....	61 C.	200 F.....	140 F.
150 C.....	65 C.	300 F.....	149 F.
200 C.....	71 C.	400 F.....	160 F.
250 C.....	78 C.	500 F.....	175 F.
300 C.....	87 C.	600 F.....	195 F.
350 C.....	99 C.	700 F.....	220 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Open Flash <sup>a</sup>		A.S.T.M. Gravity <sup>b</sup>	
A.S.T.M. Thermometer...	11C - 42	11F - 42	12C - 39	12F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-6 to +400 C.	+20 to +760 F.	-20 to +102 C.	-5 to +215 F.
Subdivisions.....	2 C.	5 F.	0.2 C.	0.5 F.
Total Length.....	303 to 307 mm.		403 to 409 mm.	
Stem Diameter.....	6.0 to 7.0 mm.		7.0 to 8.0 mm.	
Bulb Diameter.....	not greater than stem		6.0 to 7.0 mm.	
Bulb Length.....	not over 10 mm.		14 to 19 mm.	
Bottom of Bulb to Graduation Line at..... Distance.....	-6 C. 40 to 50 mm.	+20 F.	-20 C. 32 to 51 mm.	-5 F.
Top of Thermometer to Graduation Line at..... Distance.....	+400 C. 30 to 45 mm.	+760 F.	+102 C. 25 to 45 mm.	+215 F.
Top Finish.....	red glass ring	red glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	10 C.	10 F.	1 C.	1 F.
Graduations Numbered at Each Multiple of.....	10 C.	20 F.	2 C.	5 F.
Immersion (See Section 6)	25 mm.	1 in.	total	total
Special Marking on Thermometer (See Section 7).	25-MM IMM ASTM OPEN FLASH	1-IN IMM ASTM OPEN FLASH	ASTM GR	ASTM GR
Scale Error at any point up to..... when standardized, shall not exceed....	372 C. 1 C.	700 F. 2.5 F.	0.1 C.	0.25 F.
Standardization.....	c	c	ice point and every 15 or 20 C.	ice point and every 30 or 40 F.
Test for Permanency of Range (See Section 10)...	subject to 360 to 370 C. for 24 hr.	subject to 680 to 700 F. for 24 hr.	....	....
Marking on Case.....	A.S.T.M. Open Flash, -6 to +400 C.	A.S.T.M. Open Flash, +20 to +760 F.	A.S.T.M. Gr., -20 to +102 C.	A.S.T.M. Gr., -5 to +215 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Methods D 92 and D 243, except for the revision adopted in 1942.

<sup>b</sup> These requirements were previously specified in A.S.T.M. Method D 287.

<sup>c</sup> The thermometer shall be standardized at the ice point and at intervals of approximately 50 C. or 100 F. for 25-mm. or 1-in. immersion and for the following temperatures of the emergent mercury column:

Thermometer Reading	Average Temperature of Emergent Mercury Column	Thermometer Reading	Average Temperature of Emergent Mercury Column
100 C.....	44 C.	200 F.....	110 F.
150 C.....	54 C.	300 F.....	129 F.
200 C.....	64 C.	400 F.....	150 F.
250 C.....	77 C.	500 F.....	175 F.
300 C.....	91 C.	600 F.....	205 F.
350 C.....	108 C.	700 F.....	240 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Loss on Heat <sup>a</sup>		A.S.T.M. Paraffin Wax Melting Point <sup>b</sup>	
A.S.T.M. Thermometer.....	13C - 39		14C - 39	14F - 39
Liquid.....	mercury		mercury	mercury
Filling above Liquid.....	nitrogen gas		nitrogen gas	nitrogen gas
Temperature Range.....	155 to 170 C.		38 to 82 C.	100 to 180 F.
Subdivisions.....	0.5 C.		0.1 C.	0.2 F.
Total Length.....	150 to 154 mm.		365 to 371 mm.	
Stem Diameter.....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	5.0 to 6.0 mm.		not greater than stem	
Bulb Length.....	10 to 15 mm.		not over 28 mm.	not over 28 mm.
Bottom of Bulb to Graduation Line at Distance.....	0 C. <sup>f</sup> 33 to 38 mm.	155 C. 70 to 80 mm.	38 C. 105 to 115 mm.	100 F.
Top of Thermometer to Graduation Line at Distance.....	170 C. 20 to 30 mm.		82 C. 25 to 40 mm.	180 F.
Contraction Chamber.....	e		d	d
Expansion Chamber shall permit heating to.....	e		100 C.	212 F.
Top Finish.....	glass ring		plain	plain
Longer Graduation Lines at Each.....	1 C.		0.5 C.	1.0 F.
Graduations Numbered at Each Multiple of.....	5 C.; and at 0 C. and 163 C.		1 C.	2 F.
Immersion (See Section 6).....	total		79 mm.	3¼ in.
Special Marking on Thermometer (See Section 7).	ASTM LOSS ON HEAT		ASTM PARAFFIN MP 79-MM IMM	ASTM PARAFFIN MP 3¼-IN IMM
Scale Error at any point when standardized shall not exceed.....	0.5 C.		0.1 C.	0.2 F.
Standardization.....	at three points including 163 C. for total immersion		every 10 C. for 79-mm. immersion, and for avg. temp. 25 C. of emergent stem	every 20 F. for 3¼-in. immersion, and for avg. temp. 77 F. of emergent stem
Marking on Case.....	A.S.T.M. Loss on Heat, 155 to 170 C.		A.S.T.M. Paraffin M.P., 38 to 82 C.	A.S.T.M. Paraffin M.P., 100 to 180 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Method D 6, except for the editorial changes made in 1942.

<sup>b</sup> These requirements were previously specified in A.S.T.M. Methods D 87 and D 127.

<sup>c</sup> The contraction chamber shall be of the long, narrow type; the top shall be not more than 65 mm. above the bottom of the bulb.

<sup>d</sup> The top of the contraction chamber shall be not more than 41 mm. above the bottom of the bulb.

<sup>e</sup> The expansion chamber shall permit heating the thermometer at least 25 C. above the highest temperature on the scale.

<sup>f</sup> At the ice point the thermometer shall be graduated from - 1 C. to + 1 C. in 0.5 C. divisions.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Low Softening Point <sup>a</sup>		A.S.T.M. High Softening Point <sup>b</sup>	
	A.S.T.M. Thermometer...			
A.S.T.M. Thermometer...	15C - 39	15F - 39	16C - 39	16F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-2 to +80 C.	+30 to +180 F.	30 to 200 C.	85 to 392 F.
Subdivisions.....	0.2 C.	0.5 F.	0.5 C.	1 F.
Total Length.....	378 to 384 mm.		378 to 384 mm.	
Stem Diameter.....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	4.5 to 5.5 mm.		4.5 to 5.5 mm.	
Bulb Length.....	9.0 to 14 mm.		9.0 to 14 mm.	
Bottom of Bulb to Graduation Line at.....	0 C.	32 F.	30 C.	85 F.
Distance.....	75 to 90 mm.		75 to 90 mm.	
Top of Thermometer to Graduation Line at.....	80 C.	176 F.	200 C.	392 F.
Distance.....	30 to 45 mm.		30 to 45 mm.	
Expansion Chamber shall permit heating to at least.....	130 C.	270 F.	250 C.	482 F.
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	1 C.	1 F.	1 C.	5 F.
Graduations Numbered at Each Multiple of.....	2 C.	5 F.	5 C.	10 F.
Immersion.....	total	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM LOW SP	ASTM LOW SP	ASTM HIGH SP	ASTM HIGH SP
Scale Error at any point when standardized shall not exceed.....	0.2 C.	0.4 F.	0.3 C.	0.5 F.
Standardization.....	ice point, and every 20 C.	ice point, and every 40 F.	every 40 C.	every 70 F.
Marking on Case.....	A.S.T.M. Low S.P., -2 to +80 C.	A.S.T.M. Low S.P., +30 to +180 F.	A.S.T.M. High S.P., 30 to 200 C.	A.S.T.M. High S.P., 85 to 392 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Methods D 36, D 61, D 139, and E 28.

<sup>b</sup> These requirements were previously specified in A.S.T.M. Method D 36, except for the temperature range, and in Method E 28.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Saybolt Viscosity <sup>a</sup>		A.S.T.M. Saybolt Viscosity	A.S.T.M. Saybolt Viscosity <sup>a</sup> A.S.T.M. Reid Vapor Pressure <sup>e</sup>
A.S.T.M. Thermometer...	17 C - 39	17 F - 39	18 C - 39	18 F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range..... For Tests at.....	19 to 27 C. 21.1 and 25 C.	66 to 80 F. 70 and 77 F.	34 to 42 C. 37.8 C.	94 to 108 F. 100 F.
Subdivisions.....	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Total Length.....	252 to 256 mm.		252 to 256 mm.	
Stem Diameter <sup>f</sup> .....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	not less than 5.0 mm. not greater than stem		not less than 5.0 mm. not greater than stem	
Bulb Length.....	25 to 35 mm.		25 to 35 mm.	
Bottom of Bulb to Gradu- ation Line at..... Distance.....	19 C. 135 to 150 mm.	66 F.	34 C. 135 to 150 mm.	94 F.
Top of Thermometer to Graduation Line at..... Distance.....	27 C. 20 to 35 mm.	80 F.	42 C. 20 to 35 mm.	108 F.
Contraction Chamber ....	b	b	b	b
Expansion Chamber.....	c	c	c	c
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	0.5 C.	1 F.	0.5 C.	1 F.
Graduations Numbered at Each Multiple of.....	1 C.	2 F.	1 C.	2 F.
Immersion.....	total	total	total	total
Special Marking on Ther- mometer (See Section 7)	ASTM SAYBOLT VIS 21.1 AND 25 C <sup>d</sup>	ASTM SAYBOLT VIS 70 AND 77 F <sup>d</sup>	ASTM SAYBOLT VIS 37.8 C <sup>d</sup>	ASTM SAYBOLT VIS 100 F <sup>d</sup>
Scale Error at any point shall not exceed .....	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Standardization.....	total immersion <sup>g</sup>	total immersion <sup>g</sup>	total immersion <sup>g</sup>	total immersion <sup>g</sup>
Marking on Case.....	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 19 to 27 C.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 66 to 80 F.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 34 to 42 C.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 94 to 108 F. or A.S.T.M. Reid Vapor Pressure Thermom- eter

<sup>a</sup> These requirements were previously specified in A.S.T.M. Method D 88.<sup>b</sup> The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. above the bottom of the bulb. The mercury shall stand in the contraction chamber at 0 C. (32 F.).<sup>c</sup> The expansion chamber shall permit heating the thermometer 50 C. (90 F.) above the highest temperature on the scale, and in all cases shall permit heating to 100 C. (212 F.).<sup>d</sup> The test temperatures shall be shown in full at the appropriate points on the scale and the graduations corresponding to these points and the numbers shall be in red.<sup>e</sup> Correction for emergent stem shall not be applied.<sup>f</sup> The stem shall be made with an enlargement not less than 4.0 nor more than 7.0 mm. in length, having a diameter 2.0 to 3.0 mm. greater than that of the stem, the bottom of the enlargement being 114 mm. above the bottom of the bulb.<sup>g</sup> These requirements were previously specified in A.S.T.M. Method D 523.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Saybolt Viscosity <sup>a</sup>		A.S.T.M. Saybolt Viscosity <sup>a</sup>	
A.S.T.M. Thermometer.....	19C - 39	19F - 39	20C - 39	20F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	49 to 57 C.	120 to 134 F.	57 to 65 C.	134 to 148 F.
For Tests at.....	50 and 54.4 C.	122 and 130 F.	60 C.	140 F.
Subdivisions.....	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Total Length.....	252 to 256 mm.		252 to 256 mm.	
Stem Diameter <sup>f</sup> .....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	not less than 5.0 mm. not greater than stem		not less than 5.0 mm. not greater than stem	
Bulb Length.....	25 to 35 mm.		25 to 35 mm.	
Bottom of Bulb to Gradu- ation Line at.....	49 C.	120 F.	57 C.	134 F.
Distance.....	135 to 150 mm.		135 to 150 mm.	
Top of Thermometer to Graduation Line at.....	57 C.	134 F.	65 C.	148 F.
Distance.....	20 to 35 mm.		20 to 35 mm.	
Contraction Chamber....	b	b	b	b
Expansion Chamber.....	c	c	c	c
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	0.5 C.	1 F.	0.5 C.	1 F.
Graduations Numbered at Each Multiple of.....	1 C.	2 F.	1 C.	2 F.
Immersion.....	total	total	total	total
Special Marking on Ther- mometer (See Section 7)	ASTM SAYBOLT VIS 50 AND 54.4 C <sup>d</sup>	ASTM SAYBOLT VIS 122 AND 130F <sup>d</sup>	ASTM SAYBOLT VIS 60 C <sup>d</sup>	ASTM SAYBOLT VIS 140 F <sup>d</sup>
Scale Error at any point shall not exceed.....	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Standardization.....	total immersion <sup>e</sup>	total immersion <sup>e</sup>	total immersion <sup>e</sup>	total immersion <sup>e</sup>
Marking on Case.....	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 49 to 57 C.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 120 to 134 F.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 57 to 65 C.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 134 to 148 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Standard Method D 88.

<sup>b</sup> The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. above the bottom of the bulb. The mercury shall stand in the contraction chamber at 0 C. (32 F.).

<sup>c</sup> The expansion chamber shall permit heating the thermometer 50 C. (90 F.) above the highest temperature on the scale, and in all cases shall permit heating to 100 C. (212 F.).

<sup>d</sup> The test temperatures shall be shown in full at the appropriate points on the scale and the graduations corresponding to these points and the numbers shall be in red.

<sup>e</sup> Correction for emergent stem shall not be applied.

<sup>f</sup> The stem shall be made with an enlargement not less than 4.0 nor more than 7.0 mm. in length, having a diameter 2.0 to 3.0 mm. greater than that of the stem, the bottom of the enlargement being 114 mm. above the bottom of the bulb.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Saybolt Viscosity <sup>a</sup>		A.S.T.M. Saybolt viscosity <sup>a</sup>	
A.S.T.M. Thermometer...	21C - 39	21F - 39	22C - 39	22F - 39
Liquid.....	mercury	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range..... For Tests at.....	79 to 87 C. 82.2 C.	174 to 188 F. 180 F.	95 to 103 C. 98.9 C.	204 to 218 F. 210 F.
Subdivisions.....	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Total Length.....	252 to 256 mm.		252 to 256 mm.	
Stem Diameter <sup>f</sup> .....	6.0 to 7.0 mm.		6.0 to 7.0 mm.	
Bulb Diameter.....	not less than 5.0 mm. not greater than stem		not less than 5.0 mm. not greater than stem	
Bulb Length.....	25 to 35 mm.		25 to 35 mm.	
Bottom of Bulb to Gradu- ation Line at..... Distance.....	79 C. 135 to 150 mm.	174 F.	95 C. 135 to 150 mm.	204 F.
Top of Thermometer to Graduation Line at.... Distance.....	87 C. 20 to 35 mm.	188 F.	103 C. 20 to 35 mm.	218 F.
Contraction Chamber....	b	b	b	b
Expansion Chamber.....	c	c	c	c
Top Finish.....	glass ring	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	0.5 C.	1 F.	0.5 C.	1 F.
Graduations Numbered at Each Multiple of.....	1 C.	2 F.	1 C.	2 F.
Immersion.....	total	total	total	total
Special Marking on Ther- mometer (See Section 7).	ASTM SAYBOLT VIS 82.2 C <sup>d</sup>	ASTM SAYBOLT VIS 180 F <sup>d</sup>	ASTM SAYBOLT VIS 98.9 C <sup>d</sup>	ASTM SAYBOLT VIS 210 F <sup>d</sup>
Scale Error at any point shall not exceed.....	0.1 C.	0.2 F.	0.1 C.	0.2 F.
Standardization.....	total immersion <sup>e</sup>	total immersion <sup>e</sup>	total immersion <sup>e</sup>	total immersion <sup>e</sup>
Marking on Case.....	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 79 to 87 C.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 174 to 188 F.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 95 to 103 C.	A.S.T.M. Saybolt Viscosimeter Ther- mometer, 204 to 218 F.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Standard Method D 88.

<sup>b</sup> The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. above the bottom of the bulb. The mercury shall stand in the contraction chamber at 0 C. (32 F.).

<sup>c</sup> The expansion chamber shall permit heating the thermometer 50 C. (90 F.) above the highest temperature on the scale, and in all cases shall permit heating to 100 C. (212 F.).

<sup>d</sup> The test temperatures shall be shown in full at the appropriate points on the scale and the graduations corresponding to these points and the numbers shall be in red.

<sup>e</sup> Correction for emergent stem shall not be applied.

<sup>f</sup> The stem shall be made with an enlargement not less than 4.0 nor more than 7.0 mm. in length, having a diameter 2.0 to 3.0 mm. greater than that of the stem, the bottom of the enlargement being 114 mm. above the bottom of the bulb.



TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Engler Viscosity <sup>a, b</sup>	A.S.T.M. Engler Viscosity <sup>a, b</sup>	A.S.T.M. Engler Viscosity <sup>a, b</sup>
A.S.T.M. Thermometer.....	23C - 39	24C - 39	25C - 39
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	18 to 28 C.	39 to 54 C.	95 to 105 C.
Subdivisions.....	0.2 C.	0.2 C.	0.2 C.
Total Length.....	200 to 207 mm.	225 to 232 mm.	200 to 207 mm.
Stem Diameter.....	5.5 to 6.5 mm.	5.5 to 6.5 mm.	5.5 to 6.5 mm.
Bulb Diameter.....	5.5 to 6.5 mm.	5.5 to 6.5 mm.	5.5 to 6.5 mm.
Bulb Length.....	13 to 19 mm.	13 to 19 mm.	13 to 19 mm.
Bottom of Bulb to Graduation Line at..... Distance.....	18 C. 108 to 118 mm.	39 C. 108 to 118 mm.	95 C. 108 to 118 mm.
Top of Thermometer to Graduation Line at..... Distance.....	28 C. 30 to 40 mm.	54 C. 30 to 40 mm.	105 C. 30 to 40 mm.
Contraction Chamber.....	c	c	c
Expansion Chamber.....	d	d	d
Top Finish.....	glass button	glass button	glass button
Longer Graduation Lines at Each.....	1 C.	1 C.	1 C.
Graduations Numbered at Each Multiple of.....	2 C.	2 C.	2 C.
Immersion (See Section 6).....	90 mm.	90 mm.	90 mm.
Special Marking on Thermometer (See Section 7).....	Full figures at 25 C. ASTM ENGLER <sup>c</sup> 90-MM IMM <sup>c</sup>	Full figures at 40 and 50 C. ASTM ENGLER <sup>c</sup> 90-MM IMM <sup>c</sup>	Full figures at 100 C ASTM ENGLER <sup>c</sup> 90-MM IMM <sup>c</sup>
Scale Error at any point shall not exceed.....	0.1 C.	0.1 C.	0.1 C.
Standardization.....	90-mm. immersion	90-mm. immersion	90-mm. immersion
Marking on Case.....	A.S.T.M. Engler Vis- cosimeter Ther- mometer, 18 to 28 C.	A.S.T.M. Engler Vis- cosimeter Ther- mometer, 39 to 54 C.	A.S.T.M. Engler Vis- cosimeter Ther- mometer, 95 to 105 C.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Standard D 300.

<sup>b</sup> The thermometer shall be mounted in a brass ferrule consisting of a tubular bushing 8.0 mm. in outside diameter with a flanged head approximately 12 mm. in diameter so that the upper extremity of the 8-mm. diameter is located 90 mm. from the bottom of the bulb.

<sup>c</sup> The contraction chamber shall be of the long, narrow type; the top shall be not more than 60 mm. above the bottom of the bulb. The mercury shall stand approximately in the middle of the chamber at 0 C.

<sup>d</sup> The expansion chamber shall permit heating the thermometer 50 C. above the highest temperature on the scale and in all cases to permit heating to 100 C.

<sup>e</sup> To be etched on the glass stem above the brass ferrule.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Stability Test of Soluble Nitrocellulose <sup>a,b</sup>	A.S.T.M. Turpen- tine Distillation <sup>c</sup>	A.S.T.M. Titer Test
A.S.T.M. Thermometer.....	26C - 42	27C - 42	36C - 42
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	....	nitrogen gas	nitrogen gas
Temperature Range.....	130 to 140 C.	147 to 182 C.	-2 to +68 C.
Subdivisions.....	0.1 C.	0.5 C.	0.2 C.
Total Length.....	18 ± ¼ in. <sup>e</sup>	291 to 296 mm.	385 to 390 mm.
Stem Diameter.....	⅜ ± ⅛ in.	6.0 to 7.0 mm.	6.0 to 7.0 mm. <sup>d</sup>
Bulb Diameter.....	....	4.0 to 5.5 mm.	5.5 to 7.0 mm. <sup>e</sup>
Bulb Length.....	2⅝ ± ¼ in.	10 to 15 mm.	15 to 25 mm.
Bottom of Bulb to Graduation Line at.....	130 C.	147 C.	-2 C.
Distance.....	8⅝ ± ¼ in.	100 to 115 mm. <sup>f</sup>	50 to 60 mm.
Top of Thermometer to Graduation Line at.....	<sup>g</sup>	182 C.	68 C.
Distance.....	<sup>g</sup>	30 to 45 mm. <sup>f</sup>	20 to 35 mm.
Contraction Chamber.....	yes	<sup>h</sup>	no
Expansion Chamber.....	<sup>i</sup>	permit heating to 230 C.	<sup>j</sup>
Top Finish.....	....	glass ring	glass ring
Longer Graduation Lines at Each.....	0.5 C.	1 C.	1 C.
Graduations Numbered at.....	each 1 C. and 130, 135, and 140 C.	each 2 C. (even)	each 2 C.
Immersion (See Section 6).....	total	76 mm.	45 mm.
Special Marking on Thermometer (See Section 7) ..	ASTM STAB NC	ASTM TURP DIST 76-MM IMM	ASTM: FAC TITER TEST 45-MM IMM
Scale Error at any point when standardized shall not exceed.....	0.2 C.	0.5 C.	0.2 C.
Standardization.....	....	At three points in- cluding 155 C. and 175 C., at 3-in. im- mersion, and avg. temp. 25 C. of emergent stem.	<sup>k</sup>
Marking on Case.....	A.S.T.M. Stability Nitrocellulose Ther- mometer, 130 to 140 C.	A.S.T.M. Turpentine Distillation Ther- mometer, 147 to 182 C.	A.S.T.M.: F.A.C. Titer Test Ther- mometer -2 to +68 C.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Method D 301, except for the revisions adopted in 1942.

<sup>b</sup> Due to the application requirements for range and construction of this thermometer, it is not practicable to include reference points. Therefore, the National Bureau of Standards will issue a report instead of a certificate for this thermometer when submitted to the Bureau for test.

<sup>c</sup> The thermometer shall have a white enamel back.

<sup>d</sup> The stem may be either the plain front or lens front type. If the thermometer is of the lens front type, the cross-section of the stem shall be such that it will pass through an 8-mm. ring gage but will not enter a 5-mm. slot gage.

<sup>e</sup> In no case shall the bulb diameter be greater than the diameter of the stem.

<sup>f</sup> The graduated portion shall be not less than 131 mm.

<sup>g</sup> The distance from the 130 C. to the 140 C. graduation marks, that is, the scale length, shall be 4⅝ ± ¼ in.

<sup>h</sup> The contraction chamber shall be of the long, narrow type; the top shall be not more than 40 mm. above the bottom of the bulb. The mercury shall stand near the bottom of the chamber at 0 C.

<sup>i</sup> An expansion chamber shall be provided to accommodate expansion up to 175 C.

<sup>j</sup> The expansion chamber shall permit heating to 85 C. The length of unchanged capillary between highest graduation and expansion chamber shall be 10 mm.

<sup>k</sup> The thermometer shall be standardized at the ice point and at intervals of approximately 20 C. for 45-mm. immersion and for an average stem temperature of emergent mercury column of 25 C.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Concluded).

Name .....	Air Chamber Thermometer (A.S.T.M. Reid Vapor-Pressure Test) <sup>a</sup>	A.S.T.M. Tung <sup>b, c</sup>
A.S.T.M. Thermometer.....	31F - 44	32C - 44
Liquid.....	mercury	mercury
Filling above Liquid.....	....	nitrogen gas or hydrogen gas
Temperature Range.....	-40 or -30 F. to +120 or +130 F.	240 to 310 C.
Subdivisions.....	1 F.	2 C.
Total Length.....	254 to 305 mm.	195 to 200 mm.
Stem Diameter.....	6.0 to 7.0 mm.	6.0 to 7.0 mm.
Bulb Diameter.....	....	not greater than stem
Bulb Length.....	not over 25 mm.	not over 9 mm.
Contraction Chamber.....	none	<sup>c</sup>
Expansion Chamber.....	yes	<sup>d</sup>
Enlargement of Button.....	....	<sup>e</sup>
Bottom of Bulb to Graduation Line at.....	....	Distance to 282 C.: 77 to 79 mm. Distance to 240 C.: 46 to 52 mm. Distance to 310 C.: 94 to 104 mm.
Top of Thermometer to Graduation Line at.....	....	.....
Distance.....	....	.....
Top Finish.....	....	plain
Longer Graduation Lines at Each.....	....	10 C.
Graduations Numbered at Each Multiple of.....	....	20 C.
Immersion.....	total	total
Special Marking on Thermometer (See Section 7).....	....	ASTM TUNG
Scale Error at any point when standardized, shall not exceed.....	0.5 F.	<sup>f</sup>
Standardization.....	....	250 C. 282 C. 270 C. 300 C.
Marking on Case.....	....	A.S.T.M. Tung Oil Thermometer, 240 to 310 C.

<sup>a</sup> These requirements were previously specified in A.S.T.M. Method D 323, except for the editorial changes made in 1942.

<sup>b</sup> These requirements in part were previously specified in A.S.T.M. Specifications D 12.

<sup>c</sup> The contraction chamber shall be not less than 13 mm. below 240 C.

<sup>d</sup> The expansion chamber or capillary extension shall permit heating to 380 C.

<sup>e</sup> Enlargement of the button shall be so located on the stem that the under-side of the button shall be 150 mm. from the bottom of the bulb; diameter of button shall be approximately 3 mm. greater than diameter of the stem.

<sup>f</sup> The error at 282 C. shall not exceed 1 C. (one half scale division) The error at other points of the scale shall not exceed 2 C. (one scale division).

<sup>g</sup> Due to the application requirements for range and construction of this thermometer, it is not practicable to include reference points. Therefore, the National Bureau of Standards will issue a report instead of a certificate for this thermometer when submitted to the Bureau for test.

## *Standard Specifications for*

### SIEVES FOR TESTING PURPOSES<sup>1</sup>

WIRE CLOTH SIEVES, ROUND-HOLE AND SQUARE-HOLE  
SCREENS OR SIEVES



A.S.T.M. Designation: E 11 - 39

ADOPTED, 1939.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation E 11; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

#### Scope

1. These specifications cover woven-wire cloth sieves, round-hole screens (sieves) and square-hole perforated plate screens (sieves) for precision testing in the classification of materials according to size (mechanical analysis, fineness, and particle size determinations). The sieves covered by these specifications are intended for general use (Note 1). A method of calibrating woven wire cloth sieves is included as information in the Appendix.

NOTE 1.—Some industries may possibly require more restricted specifications for sieves for special testing purposes.

#### WOVEN WIRE CLOTH SIEVES

##### Sieve Cloth

2. (a) Wire cloth for standard sieves shall be woven (not twilled, except the

cloth of the 62-, 53-, 44-, and 37-micron sieves) from brass, bronze, or other suitable wire, and shall not be coated or plated.

(b) The average opening between the adjacent warp and the adjacent shoot wires, taken separately, shall conform to that given in column 2 of Table I, within the "permissible variation in average opening" given in column 4. Column 3 gives the approximate equivalents in inches of the basic values in millimeters given in column 2. The average diameter of the warp and of the shoot wires, taken separately, of the cloth of any given sieve shall be within the limits given in column 6 of Table I. Column 7 gives the approximate equivalents in inches of the basic values in millimeters given in column 6. The maximum width of opening between adjacent warp or shoot wires shall not exceed the nominal width of opening by more than the "permissible variation in maximum opening" given in column 5 of Table I. An exception may be made, in the case of 8-in. sieves, if the total length of all the

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Prior to their present adoption as standard, these specifications were published as tentative from 1925 to 1926. They were adopted in 1926, but withdrawn and republished as tentative from 1938 to 1939, being revised in 1939.

These specifications comprise a consolidation of the former Standard Specifications for Sieves for Testing Purposes (E 11 - 26) and for Round-Hole Screens for Testing Purposes (E 17 - 36), which specifications were accordingly discontinued in 1938.

TABLE I.—NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES.

Size or Sieve Designation	Sieve Opening		Permissible Variations in Average Opening, per cent	Permissible Variations in Maximum Opening, <sup>a</sup> per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx. equivalents)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
COARSE SERIES						
(4.24-in.)**	107.6	4.24	±2	+3	5.6 to 9.7	0.220 to 0.380
4-in.	101.6	4.00	±2	+3	5.6 to 9.7	0.220 to 0.380
3½-in.	88.9	3.50	±2	+3	5.3 to 9.3	0.210 to 0.365
3-in.	76.2	3.00	±2	+3	4.8 to 8.1	0.190 to 0.320
2½-in.	63.5	2.50	±2	+3	4.4 to 7.1	0.175 to 0.280
(2.12-in.)**	53.8	2.12	±2	+3	4.1 to 6.2	0.160 to 0.245
2-in.	50.8	2.00	±2	+3	4.1 to 6.2	0.160 to 0.245
1½-in.	44.4	1.75	±2	+3	3.8 to 5.7	0.150 to 0.225
1¼-in.	38.1	1.50	±2	+3	3.7 to 5.3	0.145 to 0.210
1½-in.	31.7	1.25	±2	+3	3.5 to 4.8	0.140 to 0.190
(1.06-in.)**	26.9	1.06	±3	+5	3.43 to 4.50	0.135 to 0.177
1-in.	25.4	1.00	±3	+5	3.43 to 4.50	0.135 to 0.177
¾-in.	22.2	0.875	±3	+5	3.23 to 4.22	0.127 to 0.166
¾-in.	19.1	0.750	±3	+5	3.10 to 3.91	0.122 to 0.154
½-in.	15.9	0.625	±3	+5	2.74 to 3.43	0.108 to 0.135
(0.530-in.)**	13.4	0.530	±3	+5	2.39 to 3.10	0.094 to 0.122
½-in.	12.7	0.500	±3	+5	2.39 to 3.10	0.094 to 0.122
¼-in.	11.1	0.438	±3	+5	2.23 to 2.84	0.088 to 0.112
¼-in.	9.52	0.375	±3	+5	2.11 to 2.59	0.083 to 0.102
¼-in.	7.93	0.312	±3	+5	1.85 to 2.36	0.073 to 0.093
(0.265-in.)**	6.73	0.265	±3	+5	1.60 to 2.11	0.063 to 0.083
¼-in. (No. 3)	6.35	0.250	±3	+5	1.60 to 2.11	0.063 to 0.083
FINE SERIES <sup>b</sup>						
5660 micron (No. 335)	5.66	0.223	±5	+10	1.28 to 1.90	0.050 to 0.075
4760 micron (No. 4)	4.76	0.187	±5	+10	1.14 to 1.68	0.045 to 0.066
4000 micron (No. 5)	4.00	0.157	±5	+10	1.00 to 1.47	0.039 to 0.058
3360 micron (No. 6)	3.36	0.132	±5	+10	0.87 to 1.32	0.034 to 0.052
2830 micron (No. 7)	2.83	0.111	±5	+10	0.80 to 1.20	0.031 to 0.047
2380 micron (No. 8)	2.38	0.0937	±5	+10	0.74 to 1.10	0.0291 to 0.0433
2000 micron (No. 10)	2.00	0.0787	±5	+10	0.68 to 1.00	0.0268 to 0.0394
1680 micron (No. 12)	1.68	0.0661	±5	+10	0.62 to 0.90	0.0244 to 0.0354
1410 micron (No. 14)	1.41	0.0555	±5	+10	0.56 to 0.80	0.0220 to 0.0315
1190 micron (No. 16)	1.19	0.0469	±5	+10	0.50 to 0.70	0.0197 to 0.0276
1000 micron (No. 18)	1.00	0.0394	±5	+15 <sup>a</sup>	0.43 to 0.62	0.0169 to 0.0244
840 micron (No. 20)	0.84	0.0331	±5	+15 <sup>a</sup>	0.38 to 0.55	0.0150 to 0.0217
710 micron (No. 25)	0.71	0.0280	±5	+15 <sup>a</sup>	0.33 to 0.48	0.0130 to 0.0189
590 micron (No. 30)	0.59	0.0232	±5	+15 <sup>a</sup>	0.29 to 0.42	0.0114 to 0.0165
500 micron (No. 35)	0.50	0.0197	±5	+15 <sup>a</sup>	0.26 to 0.37	0.0102 to 0.0146
420 micron (No. 40)	0.42	0.0165	±5	+25 <sup>a</sup>	0.23 to 0.33	0.0091 to 0.0130
350 micron (No. 45)	0.35	0.0138	±5	+25 <sup>a</sup>	0.20 to 0.29	0.0079 to 0.0114
297 micron (No. 50)	0.297	0.0117	±5	+25 <sup>a</sup>	0.170 to 0.253	0.0067 to 0.0100
250 micron (No. 60)	0.250	0.0098	±5	+25 <sup>a</sup>	0.149 to 0.220	0.0059 to 0.0087
210 micron (No. 70)	0.210	0.0083	±5	+25 <sup>a</sup>	0.130 to 0.187	0.0051 to 0.0074
177 micron (No. 80)	0.177	0.0070	±6	+40 <sup>a</sup>	0.114 to 0.154	0.0045 to 0.0061
149 micron (No. 100)	0.149	0.0059	±6	+40 <sup>a</sup>	0.096 to 0.125	0.0038 to 0.0049
125 micron (No. 120)	0.125	0.0049	±6	+40 <sup>a</sup>	0.079 to 0.103	0.0031 to 0.0041
105 micron (No. 140)	0.105	0.0041	±6	+40 <sup>a</sup>	0.063 to 0.087	0.0025 to 0.0034
88 micron (No. 170)	0.088	0.0035	±6	+40 <sup>a</sup>	0.054 to 0.073	0.0021 to 0.0029
74 micron (No. 200)	0.074	0.0029	±7	+60 <sup>a</sup>	0.045 to 0.061	0.0018 to 0.0024
62 micron (No. 230)	0.062	0.0024	±7	+90 <sup>a</sup>	0.039 to 0.052	0.0015 to 0.0020
53 micron (No. 270)	0.053	0.0021	±7	+90 <sup>a</sup>	0.035 to 0.046	0.0014 to 0.0018
44 micron (No. 325)	0.044	0.0017	±7	+90 <sup>a</sup>	0.031 to 0.040	0.0012 to 0.0016
37 micron (No. 400)	0.037	0.0015	±7	+90 <sup>a</sup>	0.023 to 0.035	0.0009 to 0.0014

\*\* The five sieves marked in the first column with a double asterisk (\*\*) may be used instead of the 4-in., 2-in., 1-in., ½-in., and ¼-in. sieves when it is desired to have a series of sieves nesting with the Fine Series and continuing that series with the  $\sqrt{2}:1$  ratio. All of the other sieves listed above are in a  $\sqrt{2}:1$  ratio with the Fine Series within the limit of the specified permissible variations. Care should be taken in designating the five sieves marked with the double asterisk; they should not be designated as 4-in., 2-in., 1-in., ½-in., and ¼-in., but as 4.24-in., 2.12-in., 1.06-in., 0.530-in., and 0.265-in. (or by the manufacturer's nominal values, for example, for the last three 1.050-in., 0.525-in., and 0.263-in.).

<sup>a</sup> For sieves from the 1000-micron (No. 18) to the 37-micron (No. 400) size, inclusive, not more than 5 per cent of the openings shall exceed the nominal opening by more than one-half of the permissible variation in maximum opening.

<sup>b</sup> See Note 2, Section 2.

portions of rows of openings exceeding this maximum width is less than 4 in. in both the warp and the shoot directions, considered separately, and provided that the sieve is not rejected under Paragraph (d). For sieves from the 1000-micron (No. 18) to the 37-micron (No. 400) size, inclusive, not more than 5 per cent of the openings shall exceed the nominal opening by more than one-half of the permissible variation in maximum opening.

(c) Both the warp and shoot wires shall be crimped in such a manner that they will be rigid when in use.

(d) There shall be no punctures or other obvious defects in the cloth.

NOTE 2.—The micron designation of the fine sieve series represents a strong trend among users of precision sieves toward the use of the micron terminology in reporting particle sizes. The openings of successive sieves of the fine series progress in the ratio  $\sqrt{2} : 1$ , and in selecting sieves from this series it is customary to take each sieve in a given range, every alternate sieve, or every fourth sieve.

#### Standard 8-in. Sieve Frames

3. (a) *Sieve Frames for Coarse Series.*—Sieves of the coarse series having nominal openings of less than 1 in. may have frames of the standard 8-in. size (see Section 3 (b)) or may have larger frames as may be specified in individual cases. Frames for sieves of the coarse series having nominal openings of 1 in. or more may be made of either hardwood or metal and may be square, rectangular, or circular, as specified, a size larger than 8-in. being recommended. Frames, covers, and pans of the standard 8-in. size shall be made of brass, unless otherwise specified.

NOTE 3: *Special Sieves.*—These specifications do not preclude the use of special sieves for special purposes, as for example sieves having a diameter other than 8 in. or the nesting sieves for field use. When such sieves are used, in place of the standard 8-in. sieve, the cloth of the sieves should be required to conform to

these specifications. The use of other than standard 8-in. sieves where these standard sieves could be used should be discouraged, as the results are not necessarily comparable.

(b) *Sieve Frames for Fine Series.*—Frames for all sieves of the fine series shall be the standard 8-in. size, except that frames 3 in. in diameter may be used in the case of sieves No. 100 and finer, used primarily in the testing of paint pigments. The standard frames shall be circular, 8 in. (20.32 cm.) in diameter. The height of the sieve from the top of the frame to the cloth shall be either about 2 in. (5 cm.), or 1 in. (2.5 cm.). Sieves having a height of 2 in. (5 cm.) shall be designated as full-height sieves; those having a height of 1 in. (2.5 cm.) as half-height sieves. The permissible variation on the mean inside diameter  $\frac{3}{16}$  in. below the top of the sieve shall be plus  $\frac{1}{32}$  in. The bottom of the sieve or "sieve skirt" shall be so constructed as to have an easy sliding fit in any sieve conforming to the above permissible variations and in no case shall this outside diameter be less than 7.970 in. nor more than 8.000 in. Pans and covers shall be so made as to be interchangeable with standard sieves.

(c) *Mounting of Cloth in Frame.*—The cloth shall be mounted on the frame without distortion, looseness, or waviness. To prevent the material being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder or so made that the material will not catch.

#### Three-Inch Sieves

4. (a) Sieves 3 in. in diameter, used for testing paint pigments, shall be made from standard wire cloth No. 100 or finer. The sieve frames shall be circular, about 3 in. (7.6 cm.) in inside diameter, and shall not vary from this by more than plus or minus 0.16 in. (0.4 cm). The depth of the sieve from the top of

the frame to the cloth shall not be less than 0.75 in. (1.9 cm.).

(b) The frames shall be constructed of first quality sheet brass in such a manner as to be permanently rigid. To prevent the material being sieved from catching in the joint between the cloth and the frame, the joint shall be smoothly filled with solder or so made that the material will not catch.

### Label Marking

5. Each sieve (except the 3-in. sieve) shall bear a label marked with the following information: the designation of the sieve (the nominal size of the opening in inches for the coarse series, and the micron designation or the U. S. Standard Sieve Series Number for the fine series), the name of the series (for example, "U. S. Standard Sieve Series," "American Standard Series," or a specific manufacturer's series), the name of the manufacturer or responsible distributor, and the opening in inches and millimeters.

NOTE 4.—The requirements prescribed in Section 5 shall not be considered as requiring that the opening in inches be given twice on the labels for sieves of the Coarse Series, or that the opening in millimeters be given in addition to the micron designation on the labels for sieves of the Fine Series.

### ROUND-HOLE PLATE SIEVES (SIEVES)

#### Plates

6. Plates used in the manufacture of round-hole screens shall be made of brass, bronze, steel, or other rigid metal. Thickness of plates shall be governed by size of openings as well as screening area of screens and shall conform to the requirements prescribed in Table II.

#### Type of Frame

7. Frames for laboratory screens shall be at least 8 in. in diameter. Frames

for standard 8-in. laboratory screens shall conform to the requirements specified in Section 3 for woven wire-cloth sieves. Frames for large screens may be made of either hardwood or metal and may be square, rectangular, or circular, as specified. For screens having circular openings 1 in. in diameter or larger, frames larger than 8 in. in diameter are recommended.

TABLE II.—THICKNESS OF PLATES FOR ROUND-HOLE SCREENS.

Screening Area, sq. in.	Diameter of Opening, in	Thickness of Plate, in.	
		Minimum	Maximum
Under 100	All sizes.....	0.049	0.066
100 and over	$\frac{1}{8}$ and $\frac{1}{4}$ .....	0.049	0.066
	$\frac{1}{4}$ to $2\frac{1}{2}$ , incl.....	0.060	0.100
	$\frac{3}{4}$ and $3\frac{1}{2}$ .....	0.075	0.130
	$\frac{1}{2}$ and 5.....	0.105	0.160
	6 and 8.....	0.120	0.175

### Spacing of Openings

8. (a) Spacing of openings shall conform to the following requirements:

Nominal Diameter of Opening, in.	Nominal Width of Metal Between Adjacent Openings, in.
$\frac{1}{8}$ .....	$\frac{3}{16}$
$\frac{1}{4}$ .....	$\frac{3}{8}$
$\frac{3}{8}$ .....	$\frac{1}{2}$
$\frac{1}{2}$ .....	$\frac{5}{8}$
$\frac{3}{4}$ .....	$\frac{3}{4}$
$\frac{1}{2}$ .....	$\frac{1}{2}$
$\frac{3}{4}$ .....	$\frac{1}{2}$
1.....	$\frac{3}{4}$
$1\frac{1}{4}$ .....	$\frac{3}{4}$
$1\frac{1}{2}$ .....	$\frac{3}{4}$
2.....	$\frac{3}{4}$
$2\frac{1}{2}$ .....	$\frac{3}{4}$
3.....	$\frac{3}{4}$
$3\frac{1}{2}$ .....	$\frac{3}{4}$
4.....	$\frac{3}{4}$
5.....	1
6.....	1
8.....	1

(b) The openings shall be so arranged that their centers lie at the vertices of



triangles which are approximately equilateral within the limits given by the permissible variations in width of metal and diameter of opening.

#### Permissible Variations for Openings and Spacings

9. (a) For screens having openings  $\frac{1}{4}$  in. or less in diameter, the actual diameter of any opening shall not vary from the nominal diameter by more than plus or minus 5 per cent.

(b) For screens having openings over  $\frac{1}{4}$  in. in diameter, the actual diameter of any opening shall not vary from the nominal diameter by more than plus or minus 3 per cent.

(c) The width of metal between the adjacent openings in the screen plate shall not vary from the nominal value given in Section 8 (a) by more than plus or minus 20 per cent.

#### SQUARE-HOLE PLATE SCREENS (SIEVES)

##### Square-Hole Screens

10. Where square-hole plate screens are specified for use, the openings shall be the same as the openings of woven wire cloth sieves, but in other respects, except for the arrangement of the openings, they shall conform to the requirements for round-hole screens specified in Sections 6 to 9.

## APPENDIX

### METHOD OF CALIBRATING WOVEN WIRE CLOTH SIEVES

A1. The first test of any sieve should be to determine whether it conforms to the specifications. If a suitable standard of powdered or granular material is available for a fineness test, that test is advisable as an additional means of calibration. Pieces of unmounted sieve cloth should be tested in sections of a size suitable for mounting in the sieve frames. In some cases it may be desirable to make a detailed systematic microscopic test of the sieve, plotting the frequency of occurrence of different sizes of openings across the sieve, rejecting, if necessary, a sieve found to have an excessive non-uniformity of sizes of openings for the particular purpose for which the sieve is to be used.

A2. The diameters of the openings of the round-hole screens should be measured by means of an accurate steel rule or by other suitable means. The use of "paddle gages" (flat plug gages) or of tapered pin gages is recommended for checking these openings more precisely.

A3. To determine whether a sieve conforms to the foregoing specifications, the apparatus used may be of the general type as described below and in the National Bureau of Standards *Letter Circular 72*, July 26, 1922, and the test method may follow the procedure herein described:

The apparatus consists of a light-tight box about 40 cm. square and 1 m. in length, with a microscope mounted on one end and

a ground-glass plate 2 mm. in thickness on the other end. The source of illumination is a microscope illuminator containing a concentrated filament lamp, 6 v., 108 w., connected through a transformer to a 110-v. alternating current supply circuit. The light passes through a lens in the end of the illuminator and is focused on the objective of the microscope. After passing through the microscope it diverges to the ground glass plate which is mounted with the ground side in. A 50-cm. steel scale is mounted against the inner face of the ground-glass plate in such a way that the graduations of the scale may be seen through the glass. The position of the scale allows a direct reading on the edges of the image cast by the wire of the sieve and avoids parallax due to the thickness of the glass. By oiling the ground surface slightly, the visibility is greatly increased without diminishing the distinctness of the image.

A frame for holding the sieve is placed on a platform so arranged as to permit a lateral motion of about 8 in., and also motion at right angles for focusing. Long rods, extending to the end of the apparatus at which the observer is seated, enable the observer to move the sieve without leaving his place, the lateral motion being accomplished by means of a rack and pinion and the focusing by the use of beveled gears. If the frame were also provided with a



vertical motion, the utility of the apparatus would be increased. A green glass filter is placed between the lamp and the condensing lens. The filter relieves eye strain very considerably and practically eliminates the color bands otherwise appearing on the edges of the image.

In use, the sieve is mounted in its holder on the focusing platform, between the illuminator and the objective of the microscope, and is focused by the observer until a sharp image is seen on the ground glass. Measurements are then taken in millimeters by reading the positions on the steel scale where the two edges of the image of the wire cross it, a reading glass being sometimes used. The sieve is then moved across the field, readings being taken at several places on the cloth, until the whole diameter of the sieve has been traversed, care being taken at the same time to watch for the uniformity of spacing and to measure any excessively large openings. The sieve is then rotated through 90 deg. and the process repeated.

The magnification of the apparatus may be determined by means of a calibrated stage micrometer.

By using a microscope having a tube about 15 cm. long and an eyepiece with a magnifying power of approximately eight diameters, together with a 16-mm. objective, a magnification of about 250 diameters is obtained.

The National Bureau of Standards has found by experience that in testing sieves for conformity to standard specifications, the most reliable results are obtained by measuring the wire diameters and determining the number of wires per centimeter, and then computing the average opening. From five to ten wires, sometimes more, are measured depending upon the uniformity of diameter of the wire, the closeness of the measurements of the cloth to the limits permitted by the permissible variations, and the experience of the observer. Four measurements are made on each wire. Large openings are measured at the same time.

Magnifications found suitable and convenient at the National Bureau of Standards are:

Sieves	Magnifications
590 micron (No. 30) and coarser.....	45
500 to 250 micron (Nos. 35 to 60), incl....	90
210 to 37 micron (Nos. 70 to 400), incl....	250

Greater magnifications would be feasible if the distance from the microscope to the ground-glass plate were increased, provided the optical parts of the microscope were of sufficient quality.

In making all measurements, the readings

of the steel scale are estimated and recorded to 0.1 mm.

The most generally used method of determining the *mesh* of the sieve is by means of what is sometimes known as "picket-fence interference," also the "moire effect." For the No. 200 sieve, a glass scale is accurately graduated with 200 lines to the inch, the opaque lines being about equal in width to the space between the lines. The exact number of lines per unit length is immaterial, however, provided the graduation is uniform and the exact number per unit length is known. When a scale such as this is laid on a piece of woven wire cloth having a mesh per unit length approximating the number of lines per unit length on the scale, and a strong light is placed beneath, dark bands will appear which in a unit length are equal in number to the difference between the mesh of the cloth and the graduation of the scale. If the scale is moved length-

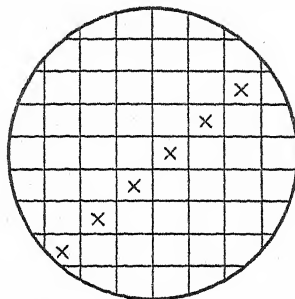


FIG. 1—Method of Selecting Openings for Plotting of Different Sizes of Openings (see Paragraph A4).

Only the indicated openings are measured.

wise, in a direction perpendicular to the lines, the dark bands will also move. If these bands move in the same direction as the motion of the scale, the mesh of the sieve is less, if the motion is in the opposite direction, the mesh is greater than the number of lines on the scale. If some bands move in one direction and some in the other, the number moving in each direction must be counted separately, and the algebraic sum taken as the number of bands. For sieves 840 micron (No. 20) to 210 micron (No. 70), inclusive, the best results are obtained with a transparent line about the width of a wire diameter ruled on an opaque background. For sieves coarser than these it is usually necessary to count the fringes by the aid of a hand lens using a steel scale as the standard. Scales should be calibrated before being used in testing sieves.

The limiting values for average opening and for maximum opening are found by multiplying

the nominal values in column 2 of Table I by the permissible variations expressed in per cent (columns 4 and 5 of Table I)—considered as exact figures followed by zeros after the decimal point—and rounding off the result to the same number of decimal places as given in column 2 of Table I.

A4. If it is desired to plot the frequency of occurrence of different sizes of openings across the sieve, 100 openings in the sieve should be measured in a diagonal direction across the sieve, then 100 openings are measured in a diagonal direction at right angles to the first. Six fields are chosen in each direction, and in any

one field the diagonal method of measurement is used as illustrated in Fig. 1.

Each opening is measured between the warp wires and also between the shoot wires; the warp readings and the shoot readings are separately tabulated and plotted.

A5. The National Bureau of Standards accepts sieves for test to determine conformity to specifications.

A6. Glass scales such as are described above may be obtained from: Bausch & Lomb Optical Co., Rochester, N. Y., and Keuffel & Esser Co., Hoboken, N. J.

### PRESENTATION OF DATA

A7. Sieve tests should be presented in tabular or graphical form in terms of the nominal sieve opening and the percentage by weight. For purposes of comparison the cumulative percentage undersize will be accepted as standard. This does not preclude the representation of percentages on individual sieves, provided the sieve interval is clearly specified as plus one sieve number and minus another, or as between two sieve apertures. Graphical repre-

sentation may include the use of logarithmic scales, probability paper, etc., to emphasize specific characteristics of shape.

A8. The presentation of data for round-hole screens should follow the same general procedure as that specified in Paragraph A7 of this Appendix for square apertures, but in all cases the term *diameter* should be applied to the size of the opening.

## Standard Methods of VERIFICATION OF TESTING MACHINES<sup>1</sup>



A.S.T.M. Designation: E 4 - 36

ADOPTED, 1936.<sup>2</sup>

Reapproved in 1942 Without Change.

This Standard of the American Society for Testing Materials is issued under the fixed designation E 4; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

### Scope

1. These methods cover procedures for the verification of testing machines by means of standard calibrating devices.

### DEFINITIONS

#### Definitions

2. (a) *Testing Machine*.—A mechanical device for applying a load (force) to a specimen.

NOTE.—Usually the magnitude of the load can be changed at the will of the operator. Many testing machines are arranged to measure the load, but this is not always the case, especially with impact machines and machines for testing ductility.

(b) *Accurate*.—A testing machine is said to be accurate if the indicated load is within the specified permissible variation from the actual load.

NOTE.—The word "accurate" applied to a testing machine shall be used without numerical values, for example, "An accurate testing machine was used for the investigation."

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Prior to their present adoption as standard, these methods were published as tentative from 1923 to 1924, being revised in 1924. They were adopted in 1924, but withdrawn and republished as tentative from 1933 to 1936, being revised in 1934, 1935, and 1936. Editorially revised and rearranged in 1939.

The accuracy of a testing machine should not be confused with *sensitiveness*. For example, a testing machine might be very sensitive, that is, it might indicate quickly and definitely small changes in the load, but, nevertheless, be very inaccurate. On the other hand, the accuracy of the results is, in general, limited by the sensitivity.

(c) *Error*.—In the case of a testing machine, the value obtained by subtracting the correct value of the quantity measured (usually load) from the indicated value as given by the testing machine.

NOTE.—The error may be positive or negative.

The word "error" shall be used with numerical values, for example, "At a load of 30,000 lb. the error of the testing machine was 15 lb."

(d) *Percentage of Error*.—In the case of a testing machine, the ratio, expressed as a percentage, of the error to the correct value of the quantity measured.

(e) *Correction*.—In the case of a testing machine, the value obtained by subtracting the indicated value from the correct value of the quantity measured.

NOTE.—The correction has the same magnitude as the error but the opposite sign. It is recommended that, except for special cases, no

corrections be used on machines tested and found to have errors within the permissible variations given in these methods.

(f) *Permissible Variation*.—In the case of a testing machine, the maximum allowable error in the value of the quantity indicated.

NOTE.—It is convenient to express permissible variation in terms of percentage of error. The numerical value of the permissible variation for a testing machine is so stated hereafter in these methods.

(g) *Loading Range*.—The loading range of a testing machine or of an apparatus for calibrating a testing machine is the range of indicated loads for which the testing machine or the calibrating apparatus gives results within permissible variations specified.

(h) *Elastic Calibration Device*.—An elastic calibration device for use in verifying the load readings of a testing machine consists of an elastic member to which loads may be applied combined with a mechanism for indicating the magnitude of deformation under load.

#### METHODS FOR VERIFYING TESTING MACHINES THAT MEASURE LOAD

#### Advantages and Limitations of Different Methods

3. Four methods of verifying testing machines are listed in this standard, as follows:

(a) *Verification by Standard Weights*.—Verification by the direct application of standard weights to the weighing mechanism of the testing machine, where practicable, is the most accurate method. Its limitations are (1) the small range of load which can be covered, (2) the nonportability of any large amount of standard weights, and (3) its nonapplicability to horizontal testing machines or to vertical testing machines whose weighing mechanisms are attached to their upper platens.

(b) *Verification by Proving Levers*.—Verification by the use of standardized proving levers loaded with standard weights ranks second in accuracy. Its limitations are (1) the fact that the range of load possible with proving levers, while greater than with standard weights, is not great enough to cover the capacity range of large testing machines, and (2) the inconvenience of transportation of proving levers and standard weights for verifying large testing machines.

(c) *Verification by Elastic Calibration Device*.—Verification by the use of an elastic calibration device ranks third in accuracy. It is free from the limitations of the methods referred to in Paragraphs (a) and (b).

(d) *Verification by Comparison Method*.—Verification by a comparison of the tensile strength of test specimens ("companion specimens") cut from the same piece of metal, is distinctly less accurate than the three methods referred to in Paragraphs (a), (b), and (c), and should be used only when none of the other methods are available. If the results of a verification test by the comparison method fail to agree with the results of a verification test by any of the other three methods, the results given by the comparison method shall be discarded.

#### Method of Applying Load

4. In the verification of a testing machine, the loads shall be applied in ascending order.

NOTE.—For any testing machine (particularly for certain types of machines in which the load-indicating device is actuated by a Bourdon pressure tube, a hydraulic (or steam-engine) indicator, or other device depending on the elastic properties of a material, or for machines in which the load is measured by measuring the pressure in a hydraulic jack), the errors observed at corresponding loads taken first by increasing the load to the test load and then by decreasing it to the test load may not agree. Testing machines are usually used under in-

creasing loads, but if a testing machine is to be used under decreasing loads it should be calibrated under decreasing loads as well as under increasing loads.

### Selection of Test Loads

5. For any loading range, the testing machine shall be verified by at least five test loads (except for testing machines designed to measure only a smaller number of definite loads, such as certain hardness testing machines). The difference between any two successive test loads shall not exceed one-third of the

along the axis of the testing machine as is possible.

NOTE.—The effect of eccentric load on the accuracy of a testing machine may be determined by calibration readings taken with proving levers or an elastic calibration device placed so that the resultant load is applied at definite distances from the axis of the machine, and the loading range determined for a series of eccentricities. In the case of testing machines in which the load reading depends on the hydrostatic pressure in a cylinder fitted with a piston, the effect of eccentricity of loading is most serious when the piston is at the extreme outward position allowable.

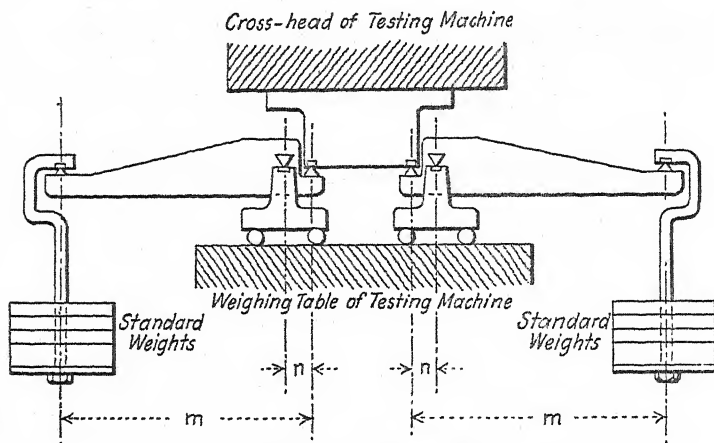


FIG. 1.—Proving Levers.

difference between the maximum and minimum test loads.

NOTE.—A testing machine may have more than one loading range. For instance, a 100,000-lb. capacity testing machine may have one loading range from 5000 lb. to 40,000 lb. and a second loading range from 55,000 lb. to 100,000 lb. Section 4 requires that the machine must be verified by at least five test loads between 5000 lb. and 40,000 lb. and also that the machine must be verified by at least five test loads between 55,000 lb. and 100,000 lb.

### Eccentric Loading

6. For the purpose of determining the loading range of a testing machine, all calibration loads shall be applied so that the resultant load shall be as nearly

### Verification by Standard Weights

#### Procedure

7. Standard metal weights of suitable design, finish, and adjustment shall be placed on the weighing platform of the testing machine or upon trays or other supports suspended from the load-measuring mechanism in place of the specimen. The weights shall be applied in increments and removed in the reverse order. They shall be arranged symmetrically with respect to the weighing platform, so that the center of gravity of the load lies in the vertical line through the center of the platform.

The applied load and the indicated load shall be recorded for each test load applied, and the error calculated from these data.

NOTE.—The method of verification by standard weights can be used only on vertical testing machines. The total load is limited by the size of the platform and the number of weights available. Often 20 weights of 50 lb. each are used, a total of 1000 lb.

This method of verification is seldom used for any but small testing machines, as the loading range does not include the loads for which large machines are used.

### *Verification by Standardized Proving Levers*

#### **Levers**

8. (a) The arrangement of proving levers for verifying testing machines is shown in Fig. 1. The two levers rest on supports on the weighing platform of the testing machine. These supports shall move easily in a horizontal direction, which insures that the forces at each of the knife edges shall be very nearly vertical. The inner knife edges in each lever bear against a suitable block in the movable head of the testing machine. Weight trays or hangers are suspended from each of the outer knife edges and these trays or hangers are loaded with standard weights. The increment of load put on the testing machine by the standard weights is the amount of standard weights multiplied by the lever ratio  $m/n$ , Fig. 1.

(b) The knife edges, as well as their supports, shall be of hardened tool steel. The knife edges shall be ground sharp to an angle of 90 deg. The load on any knife edge shall not exceed 7000 lb. per linear inch. The three knife edges in each lever shall be parallel, and their edges shall lie in a plane. Each lever shall have machined surfaces, in this or in some parallel plane, upon which a spirit level can be placed.

#### **Verification of Levers**

9. The lever ratio of a proving lever shall be determined by the use of at least three test loads; the amount of weights used shall not be less than the maximum load applied upon one of the arms of a lever in using the levers to verify testing machines. The proving lever shall be balanced over its center knife edge with suitable weight trays suspended from the end knife edges. Standard weights shall then be applied to the trays in three steps, corresponding approximately to 50, 75, and 100 per cent of the weights available, and the proving lever shall then be brought to a balance by the use of small weights and by observations of the freely swinging proving lever. From the weights in the two weight trays the lever ratio shall be calculated.

#### **Procedure**

10. The proving levers shall be placed symmetrically in the testing machine to be verified, and both levers shall be brought as near to a horizontal position as is feasible, after applying each increment of load, by means of the movable head of the testing machine. The testing machine shall be balanced with the levers in place and the weight trays empty. Standard weights shall be applied (or removed) in increments, half an increment in (or from) each tray. The weights shall be placed symmetrically on the weight trays, with the center of gravity of the weights over the center of the tray. The applied load and the indicated load shall be recorded for each test load applied, and the error calculated from these data.

NOTE.—The use of standardized proving levers on horizontal testing machines involves the use of bell crank levers. They require special methods of determination of lever ratio, which have not yet been codified in A.S.T.M. standards. Proving levers for vertical testing machines are now available up to 50,000-lb. capacity.



### *Verification by Means of an Elastic Calibration Device<sup>3</sup>*

#### **Design and Workmanship**

11. An elastic calibration device should be so designed and constructed that its accuracy is not in danger of being impaired by handling, shipping, or ordinary use, and so that parts subject to damage, replacement, or removal for storage and shipment can be replaced without impairing the accuracy of the device.

NOTE.—This section is nonquantitative in its requirements. It is placed in the method for the purpose of calling attention to the fact that an elastic calibration device, if improperly designed and constructed, may, like any other delicate measuring instrument, be damaged and rendered inaccurate by handling and shipping.

#### **Shackles and Bearing Blocks**

12. An elastic calibration device shall be provided with shackles, bearing blocks, or any other necessary fixtures through which the load is to be applied to the calibration device. All such fixtures should be so designed and constructed that when placed in a testing machine in reasonably good condition there shall be no variation of reading in excess of permissible variations, due to imperfections in the bearing blocks or in the motion of the head of the testing machine.

NOTE.—This section is nonquantitative in its requirements. It is placed in the method for the purpose of calling attention to the fact that poorly designed loading fixtures can render inaccurate the readings of an elastic calibration device.

#### **Graduation of Scale**

13. (a) The scale of the indicating mechanism shall be uniformly graduated. The distance between any two gradua-

tion lines, dots, or other dividing marks (or between their images, if a viewing system with a definite optical magnification is used), shall be not less than 0.035 in.

(b) The difference between the position of any graduation line and its correct position on the scale shall not exceed one twentieth of the distance between two adjacent graduation lines. For scales on a circular dial, when successive lines are set to one fixed index line, the positions of successive graduation lines nearly diametrically opposite, referred to another fixed index line shall differ from each other by (1) not more than 5 per cent of the smallest division of the dial, or (2) not more than 0.1 per cent of the minimum load in the loading range of the instrument, if (2) be larger than (1).

(c) The change of load necessary to cause a relative movement of 0.1 in. between the index line and the scale (or its image if a viewing system with a definite optical magnification is used) shall not exceed 1 per cent of the capacity load of the instrument.

#### **Label Showing Manufacturer, Number, and Capacity**

14. The manufacturer's name, the capacity load, and the serial number of the device shall be legibly marked on some part of the instrument.

#### **Temperature Equalization**

15. When using an elastic calibration device to verify the load readings of a testing machine, the device should be placed near, or preferably in, the testing machine a sufficient length of time before the test so that the device and the testing machine shall be at very nearly the same temperature.

<sup>3</sup>The term "calibration device" shall be interpreted to include solid bars, hollow bars, elastic loops, and other members whose elastic deformation can be measured.

### Elimination of Small Temperature Effects Due to Stress

16. To minimize errors due to temperature effect of stress and pseudo-elastic action, the time schedule followed when verifying an elastic device which is loaded in axial tension or compression shall be reported; and the same time schedule, within limits stated in the report, shall be followed when using the device to calibrate a testing machine.

### Primary Standards for Elastic Calibration Devices

17. (a) For loads not exceeding 100,000 lb., an elastic calibration device shall itself be calibrated by applying dead weights known to be accurate within 0.02 per cent.

(b) For loads exceeding 100,000 lb., an elastic calibration device may be calibrated by another elastic calibration device or by a combination of several elastic calibration devices, or by proving levers<sup>4</sup> and dead weights. In this case the calibrating load applied shall be known to be accurate within 0.1 per cent.

### Preliminary Cyclic Loading

18. Before taking readings for the formal verification of an elastic calibration device by means of dead weights or other primary standards, the device shall be subjected to a series of cyclic loads varying from a minimum load not greater than 2 per cent of the capacity load of the device to a maximum load not less than 5 per cent nor more than 10 per cent above the nominal capacity load.

### Routine in Calibration of Elastic Calibration Device

19. For any range of test loading which may be applied in calibrating an elastic calibration device, there shall be

applied at least eight test loads, and the difference between any two successive test loads shall not exceed one-fifth of the difference between the minimum and the maximum test loads. The series of test loads shall be applied to the elastic calibration device at least three times, and the shackles or bearing blocks shall be readjusted before each application of a series of loads.

NOTE.—If the magnitude of deformation of the elastic calibration device is measured with a self-indicating dial micrometer or dial indicator, the elastic calibration device shall be used only at the test loads for which it has itself been calibrated by comparison with a primary standard. It shall be used only when the deformation measuring apparatus is so adjusted that the no-load reading of the device is nearly equal to the no-load reading observed during the calibration of the elastic calibration device.

### Permissible Variations for Elastic Calibration Device

20. (a) For ordinary use, the loading range of an elastic calibration device shall be that range of load within which any reading under an applied load shall not differ from the average of at least three readings under the same load by more than one-fifth of 1 per cent.<sup>5</sup>

(b) The report of the verification of the elastic calibration device shall also state the loading range within which no reading varies from the average of at least three readings under the same load by more than one-tenth of 1 per cent. This range shall be designated as the *Precision Loading Range* for the device.

### Temperature Correction for Elastic Calibration Device

21. In using an elastic calibration device made of steel with not more than 5 per cent of alloying elements, a fairly accurate correction for variation in temperature may be made on the basis

<sup>4</sup> The term "proving levers" is not to be interpreted as allowing the use of the lever system of a compound lever testing machine or of a platform scale as a part of a primary standard.

<sup>5</sup> This means that the report of a standardizing laboratory on an elastic calibration device will state *within what loading range* it may be used, rather than reporting a blanket acceptance or rejection of the device. This follows the practice recommended for testing machines.



that the modulus of elasticity in tension or compression (or flexure) diminishes by 0.015 per cent for each degree Fahrenheit increase in temperature. For other metals, if the change in the modulus of elasticity with temperature is known, a correction for temperature should be applied to the readings of the instrument. During any calibration of a testing machine with an elastic calibration device, or during the calibration of the device itself, temperature shall be kept as uniform as possible.<sup>6</sup>

### *Verification by Companion Specimen Method*

#### **Description of Comparison Method**

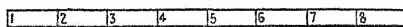
22. When the direct method of verification of a testing machine cannot be carried out (as is the case at present with horizontal testing machines, for example), or when an elastic calibration device is not available, a comparison method of verification may be used. In this method the machine to be verified is tested by comparing its indications with the corresponding readings obtained under the same conditions by the use of another testing machine, especially tested and verified, as specified in Section 23. The method of verification by comparison shall be carried out by the use of a series of companion specimens, half of which are to be tested in tension in the machine to be verified, and half of which are to be tested in tension in the especially verified testing machine which serves as a standard machine. The general provisions respecting the application of the test loads and the loading range which are outlined in the direct method of verifying testing machines (Sections 4, 5, and 6) shall apply to the verification of testing machines by the

comparison method, so far as is practicable.

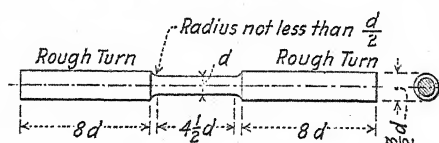
#### **Requirements for Testing Machines Used for Verifying Other Machines**

23. (a) A testing machine which is to be used as the standard for verifying other testing machines shall itself be verified by a special test, in which the verification is carried out twice. The loading range shall be determined by the permissible variation of plus or minus 1 per cent (Section 27). For the loading range established, the differences in indications found in the two tests of the standard machine shall not exceed 0.25 per cent.

(b) In verifying testing machines by the companion specimen method, corrections shall be applied to the indications of the standard machine and these corrections shall be based on the average



(a) Method of Numbering Specimens.



(b) Form of Test Specimen.

FIG. 2.—Companion Specimen.

correction obtained in the two tests used in its verification.

#### **Test Specimens**

24. (a) For each test load, eight or more tension test specimens shall be cut from soft-rolled or drawn steel and numbered consecutively, as in Fig. 2 (a).

(b) The tensile strength of the steel shall be determined by a preliminary test and the sets of specimens for the comparison test shall have such nominal cross-sectional areas as will give, approximately, the loads required. The form of the specimen shall be as shown in Fig. 2 (b). The ends may be threaded or otherwise machined to fit holders,

<sup>6</sup> Similar errors due to change of modulus of elasticity with temperature may be present in the load-indicating mechanism of a testing machine using a Bourdon gage or other device dependent on the elastic deformation of a member.

but it is recommended that those specimens shipped to another laboratory be left with cylindrical ends. The actual value of " $d$ " (Fig. 2 (b)) for each specimen shall be determined by means of a micrometer.

NOTE.—It should be noted that the standard tension test specimen, 0.5 in. in diameter and having a 2-in. gage length, satisfies the requirements for a specimen for the verification of a testing machine by the comparison method. See Fig. 3 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8) of the American Society for Testing Materials.<sup>7</sup>

### Procedure

25. (a) The specimens having odd numbers shall be tested in the machine to be verified, and those having even numbers shall be tested in the standard testing machine. The tensile strength only shall be determined.

(b) The speed of the testing machine, by which is meant the speed of the pulling head when the machine is running idle with no specimen in the machine, when testing verification specimens shall be approximately the same for both the standard testing machine and the machine being verified. The speed shall be the slowest speed that can be obtained on both machines; in any case the speed shall not exceed 1 in. per min. per inch of diameter of companion specimen.

(c) When testing companion specimens, especial care shall be taken to maintain the weighing beam of the testing machine in equilibrium as the tensile strength of the specimen is approached. It is recommended that a mark be placed at the middle of the trig loop, and that a pointer be placed on the beam for verification tests, and that the pointer be kept as nearly on the mark as possible.

### Calculation of Error

26. (a) The average tensile strength (in pounds per square inch) of the odd-numbered specimens, multiplied by the nominal area of cross-section, shall be considered a measure of the indicated load, and the average tensile strength of the even-numbered specimens, multiplied by the nominal area of cross-section, shall be considered a measure of the applied load. The error shall be calculated from these data.

(b) If the tensile strength (in pounds per square inch) of any companion specimen varies more than 1 per cent from the mean of the group with which it is tested, its tensile strength shall not be used in calculating the error of the testing machine. If more than one specimen in the odd-numbered group or more than one specimen in the even-numbered group exceeds the permissible variation in tensile strength, all the specimens shall be discarded and another set prepared.

### PERMISSIBLE VARIATIONS FOR TESTING MACHINES THAT MEASURE LOAD

#### Permissible Variation Within Loading Range of a Testing Machine

27. The error for loads within the loading range of a testing machine shall not exceed 1 per cent (Notes 1 and 2).

NOTE 1.—This means that the report of the verification of a testing machine will state *within what loading range it may be used*, rather than reporting a blanket acceptance or rejection of the machine.

NOTE 2.—In no case shall the loading range be stated as including loads below a value which is 100 times the smallest load which can be read on the testing machine. (This means that if a testing machine had graduations so spaced that it can be read to within  $\frac{1}{10}$  division and the machine is sensitive to  $\frac{1}{10}$  division it would be possible for the loading range to extend down to the load corresponding to 10 divisions. On the other hand, if the testing machine was sensitive to only 2 divisions, the

<sup>7</sup> 1946 Book of A.S.T.M. Standards, Parts I-A and I-B.

loading range could not extend below the load corresponding to 200 divisions.)

### Corrections

28. The indicated load of a testing machine shall not be corrected either by calculation or by the use of a calibration diagram to obtain values within the required permissible variation.

### TIME INTERVAL BETWEEN VERIFICATIONS

#### Time Interval Between Verifications

29. It is recommended that testing machines, when in constant use, be verified at intervals of 6 months and, when used intermittently, at intervals of 2 or 3 yr. Testing machines shall, however, be verified immediately after making repairs or adjustments of the weighing mechanism, after the testing machine has been moved (this does not apply to portable testing machines), and whenever there is reason to doubt the accuracy of the results, without regard to the time interval since the last verification.

## REPORTS AND CERTIFICATES

### Reports

30. A clear and complete report shall be prepared of each verification of a testing machine. This report shall state the method of verification used, and shall give the serial numbers and the names of manufacturers of all apparatus used in carrying out the verification. It shall state how, by whom, and when the calibration of the apparatus used in verifying the testing machine was made, the loading range, the "precision" loading range of the calibration apparatus, and the loading range of the testing machine.

### Certificates

31. A certificate giving the manufacturer's serial number and a brief description of the testing machine, the manufacturer's name, the date of verification, and the loading range shall be signed by the person responsible for the maintenance of the testing machine, and this certificate shall be posted in plain view of a person operating the testing machine.

# Standard Definitions of TERMS RELATING TO METHODS OF TESTING<sup>1</sup>



A.S.T.M. Designation: E 6 - 36

ADOPTED, 1930; REVISED, 1936.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation E 6; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Stress.**—The intensity (measured per unit area) of the internal distributed forces or components of force which resist a change in the form of a body. Stress is measured in force per unit area (pounds per square inch, kilograms per square millimeter, etc.).

**NOTE.**—In examining the definitions given in various authoritative textbooks two definitions of the term “stress” were found. Textbooks in physics and some European textbooks in mechanics of materials gave a definition substantially as above. American textbooks in engineering mechanics define stress as a *force*, measured in *pounds*. They call the intensity of force (pounds per square inch) “unit stress,” “intensity of stress,” or “fiber stress.” This use of the term stress is illustrated by the “stress sheet” of the bridge engineer which gives *forces*, measured in pounds.

In view of the fact that even in engineering textbooks the term “stress” is often used to denote intensity of force per unit of area, and in view of the fact that the physicists’ definitions involve somewhat simpler terms, the physicists’ definitions have been followed in these standards.

While it is important to have a clear defini-

tion for the term “stress” it is even more important to keep clearly in mind the *units* used in calculations and test data for materials of construction. In fact, if the units are always given, there can be no misunderstanding as to the sense in which the term “stress” is used.

There are three kinds of stress: tensile, compressive, and shearing. Flexure involves the combination of tensile stress and compressive stress. Torsion involves shearing stress.

It is customary to calculate stress on the basis of the original dimensions of the cross-section of the body.

**Strain.**—The change per unit of length in a linear dimension of a body, which change accompanies a stress. Strain is measured in inches per inch of length (millimeters per millimeter) or in per cent.

**NOTE.**—In some American engineering textbooks the term “strain” is used in the sense of total deformation and is measured in inches. Change of dimension per unit length is called “unit strain,” or “unit deformation.” As in the consideration of the term “stress,” the definitions given in textbooks in physics have been followed.

Under tensile stress or compressive stress, strain is measured along the dimension under consideration. Shearing strain is measured at right angles to the dimension under consideration. In torsion tests, which involve shearing stress, it is customary to measure

<sup>1</sup> Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Prior to adoption as standard, these definitions were published as tentative from 1923 to 1930, being revised in 1924 and 1925. Editorially revised in 1946.

the angle of twist, which may be translated into terms of strain.

**Stress-Strain Diagram.**—A diagram plotted with values of stress as ordinates and values of strain as abscissas.

**NOTE.**—The use of the term stress-strain diagram is frequently extended to cover diagrams plotted with values of applied load, or applied moment as ordinates, and with values of stretch, compression, deflection, or twist as abscissas.

Fig. 1 is an example of a stress-strain diagram.

Stress-strain diagrams are in some cases drawn directly by an autographic attachment to the testing machine. A more usual method of procedure consists in taking a series of load readings (from the weighing mechanism of the testing machine) with corresponding readings of the strain-indicating apparatus (extensometer, compressometer, deflectometer, or torsion indicator). The term "strainom-

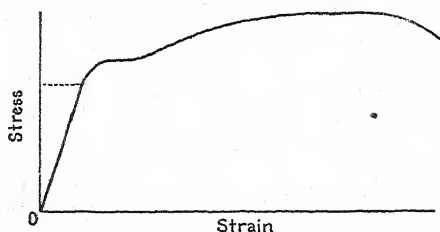


FIG. 1.—A Typical Stress-Strain Diagram with Elastic Strains Exaggerated.

eter reading" will be used to denote the reading of any strain-measuring instrument. From these readings, or from values calculated from them, there is plotted a diagram with stress-indicating values (load, flexural moment, twisting moment, or stress) as ordinates, and strain-indicating values (elongation, shortening, deflection, twist, or strain) as abscissas. In planning such a test it is necessary to decide on the increment of load or the increment of reading of strainometer to be used between successive readings.

In Fig. 2 are shown three typical stress-strain diagrams. The material for all three tests is the same, and the test specimens are all of the same size, so that the diagrams should be the same. The diagram shown in Fig. 2 (a) is plotted from points determined by taking increments of load ( $S$ ). In this diagram it is seen that owing to the shape of the curve, data for locating points between  $M$  and  $N$  are lacking. That particular portion of the curve (the "knee" of the curve)

is the part for which it is especially desirable to locate several points on the diagram.

In Fig. 2 (b) is shown a diagram plotted from points determined by taking increments of strainometer reading ( $e$ ). It will be noted that for this diagram there are located several points near the knee of the curve, and the shape of the diagram in this important region is much more definitely determined than for the curve shown in Fig. 2 (a).

The custom of choosing increments of load rather than increments of strainometer reading is quite common in tests of materials and is followed because, in general, it is easier to calculate increments of load than it is to calculate increments of strainometer reading. An estimate of the load necessary to stress the specimen up to the knee of the curve is made and some fraction (frequently one-tenth) of this value is taken as an increment. The calculation of the corresponding increment of strainometer reading is more complicated.

Sometimes the practice is followed of applying a few increments of load as determined above, and then applying load in much smaller increments ( $S'$ ) until the knee of the curve is passed. Fig. 2 (c) shows a diagram obtained in this manner. This method involves a marked increase in the number of readings necessary for a test and with unknown material there is always some danger that the knee of the curve will be reached before the use of small increments of load is begun.

The following method of choosing increments for a test is suggested: Estimate the load corresponding to the knee of the stress-strain diagram and choose a value for increment of load about one-tenth of this value. Apply this increment of load ( $S$ ) once and note the corresponding change of reading for the strainometer ( $e$ ). Then for the remainder of the test, use for the increment of strainometer reading a value which corresponds to some convenient interval on the scale of the strainometer, and which is approximately equal to ( $e$ ).

**Elastic Limit.**—The greatest stress which a material is capable of developing without a permanent deformation remaining upon complete release of the stress.

**NOTE.**—It is a matter of experience with many materials, especially with many metallic materials, that using ordinary methods of testing, the values found for elastic limit by means of observations of permanent deformation (set) after release of stress do not differ

widely from the values found for proportional limit, the latter being defined as:

**Proportional Limit.**—The greatest stress which a material is capable of developing without a deviation from the law of proportionality of stress to strain (Hooke's Law).

Since the determination of proportional limit is much more readily made than is the determination of elastic limit, it is customary to accept the proportional limit as equivalent to the elastic limit for such materials, and hence the proportional limit is frequently called the "proportional elastic limit."

For certain grades of steel a special method of determining elastic strength is recognized in the standard specifications of the American Society for Testing Materials. See the Standard Specifications for Carbon-Steel Forgings for General Industrial Use (A.S.T.M.

including the specimen in a carefully controlled laboratory test) starts as local actions and becomes measurable only after many local internal adjustments and accommodations have occurred, and after a considerable portion of the member is affected by the yielding.

The limit of usefulness of many materials, especially metals, in members subjected to approximately static loading at ordinary temperatures is therefore determined by a measurable value of plastic yielding of the material above which the material is considered to be damaged and below which the damaging effects are considered to be negligible.

The following methods are recommended for determining the yield strength of a material:

(a) *Offset Method.*—This method can be

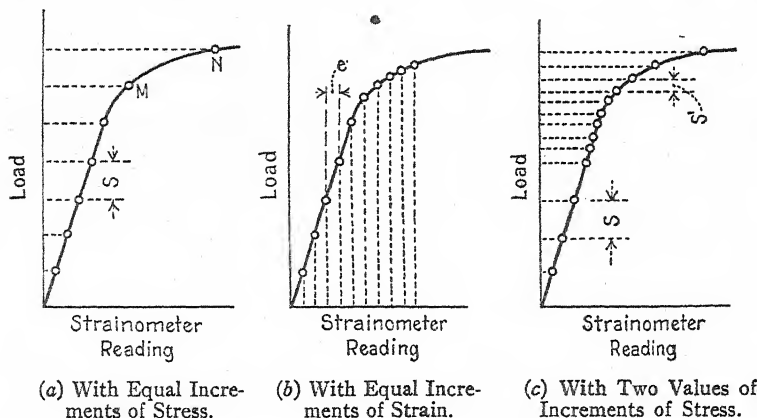


FIG. 2.—Illustrating Three Methods of Plotting Load-Deformation Curves.

Designation: A 235),<sup>3</sup> for Carbon-Steel Forgings for Locomotives and Cars (A.S.T.M. Designation: A 236),<sup>3</sup> for Alloy-Steel Forgings for General Industrial Use (A.S.T.M. Designation: A 237),<sup>3</sup> and for Alloy-Steel Forgings for Locomotives and Cars (A.S.T.M. Designation: A 238).<sup>3</sup>

**Yield Strength.**—The stress at which a material exhibits a specified limiting permanent set.

**NOTE.**—It is usually impracticable and probably impossible to determine the stress at which inelastic action in a member begins. Plastic yielding in nearly all members (in-

used, if desired, for materials having "sharp-kneed" stress-strain diagrams, but is especially adapted to materials whose stress-strain diagram in the yield range is a smooth curve of gradual curvature.

For nearly all materials, if at any point on the stress-strain diagram such as *r* in Fig. 3, the load is released, the diagram for decreasing load will follow a line *rm* approximately parallel to the initial portion *OA*, of the diagram for increasing load. The offset *Om* will then give the approximate value of the permanent set after the release of the stress *OR*. The value of this set is given in percentage strain. Thus to determine the yield strength by the "offset method," it is necessary to secure data (autographic or numerical) from which a stress-strain diagram may be drawn. Then on the stress-strain diagram

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part I-A.



(Fig. 3) lay off  $Om$  equal to the specified value of the set, draw  $mn$  parallel to  $OA$ , and thus locate  $r$ , the intersection of  $mn$  with the stress-strain diagram. Draw  $Rr$  parallel to the  $X$  axis; and then  $OR$  gives the value of the yield strength.

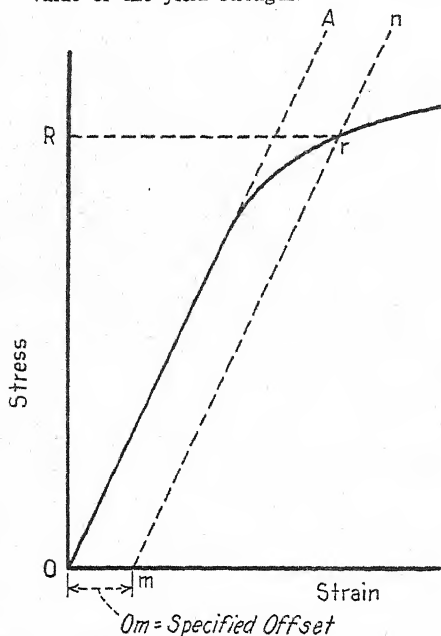


FIG. 3.—Stress-Strain Diagram.

In reporting values of yield strength obtained by this method, the specified value of "offset" used should be stated in parentheses after the term yield strength. Thus:

Yield strength (offset = 0.2 per cent) = 52,000 psi. This indicates that at a stress of 52,000 psi. the approximate permanent set of the material reached the value of 0.2 per cent (equivalent to 0.002 in. per inch of gage length).<sup>4</sup>

<sup>4</sup> Yield strength (or its equivalent) is specified as follows in the standards and tentatives of the American Society for Testing Materials:

Standards or Tentatives for:	
Alloy-Steel Pipe.....	0.2 per cent "offset"
Aluminum-Alloy Bars, Rods, Shapes, Sheet.....	
Magnesium-Base Alloy Forgings, Castings, Sheet.....	
The Alloy: Copper, 80 per cent; Tin, 10 per cent; Lead, 10 per cent.....	
Copper-Silicon Alloy Plates, Sheets, and Bars.....	0.5 per cent elon- gation under load (approx- imately equiv- alent to 0.35 per cent "off- set")
Aluminum-Bronze Castings.....	
Bronze Bearing Metals and Steam or Valve Sand Castings.....	
Composition Brass or Ounce Metal Sand Castings.....	
Naval Brass Rods for Structural Pur- poses.....	

In using this method, an extensometer reading to 0.0001 in. per inch of gage length would be sufficiently sensitive for most materials. The elastic limit and the proportional limit may be regarded as special values of the yield strength. They are the highest stresses for which the "set" and the "offset," respectively, are not measurable with the instruments used.

For any given stress the deviation (offset) from Hooke's law is only approximately equal to the set after the removal of that stress, and the approximation becomes less and less exact as the permissible deviation diminishes. This method is devised for determining a stress corresponding to a well marked plastic deformation, or set, and it is not feasible to specify a very small value for the permissible "offset"  $Om$ .

#### (b) Extension Method Under Load.—

For tests to determine the acceptance or rejection of material whose stress-strain characteristics are well known from previous tests of similar material in which stress-strain diagrams were plotted, the total strain corresponding to the stress at which the specified permanent set occurs will be known within satisfactory limits; therefore, in such tests a specified total strain may be used, and the stress on the specimen, when this total strain is reached, is the value of the yield strength. The total strain can be obtained satisfactorily by use of an extensometer reading to 0.0001 in. per inch of gage length. It is recommended that this approximate method be used only after agreement between the manufacturer and the purchaser, with the understanding that check tests be made for obtaining stress-strain diagrams for use with the offset method to settle any misunderstandings.

**Yield Point.**—The stress in a material at which there occurs a marked increase in strain without an increase in stress.

It should be noted that only materials that exhibit this unique phenomenon of yielding have a yield point. The term yield point should not be used in connection with material whose stress-strain diagram does not become horizontal or does not show an actual drop of stress with increase of strain in the region of yield. Two methods of determining yield point are in use:

(a) "*Drop of the Beam*" Method.—In this method the load is applied to the specimen at any convenient speed of testing up to one

half the specified yield point or up to one quarter the specified tensile strength, whichever is smaller, and the operator keeps the beam in balance by running out the poise at approximately a steady rate. When the yield point of the material is reached, the increase of load stops, but the operator runs the poise a trifle beyond the balance position, and the beam of the machine drops for a brief but appreciable interval of time. In a machine fitted with a self-indicating load-measuring device, there is a sudden halt of the load-indicating pointer corresponding to the drop of the beam. The load at the "halt in the gage" or the "drop of the beam" is recorded, and the corresponding stress is taken as the yield point.

(b) *Total Strain Method Using Dividers.*—In this method, frequently called the "dividers method," the observer with a pair of dividers or other suitable apparatus watches for visible elongation between two gage marks on the specimen. When visible stretch is observed, the load at that instant is noted, and the stress corresponding to the load is taken as the yield point. For the higher strength steels, a gage length of less than 8 in. is recommended.

**Tensile Strength.**—The maximum tensile stress which a material is capable of developing.

**NOTE.**—In practice, it is considered to be the maximum stress developed by a specimen

representing the material in a tension test carried to rupture, under definite prescribed conditions. Tensile strength is calculated from the maximum load carried during a tension test and the original cross-sectional area of the specimen.

**Compressive Strength.**—The maximum compressive stress which a material is capable of developing.

**NOTE.**—In the case of a material which fails in compression by a shattering fracture the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture the value obtained for compressive strength is an arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the material.

**Modulus of Elasticity.**—The ratio, within the elastic limit of a material, of stress to corresponding strain.

**NOTE.**—As there are three kinds of stress, so are there three moduli of elasticity for any material: the modulus in tension, the modulus in compression, and the modulus in shear. The value of the modulus of elasticity in tension is nearly the same, for most metals, as the value of the modulus of elasticity in compression. The value of the modulus of elasticity in shear is smaller than the value of the modulus of elasticity in tension. The modulus of elasticity is expressed in pounds per square inch (kilograms per square millimeter).



## Standard Definitions of TERMS RELATING TO SPECIFIC GRAVITY<sup>1</sup>



A.S.T.M. Designation: E 12 - 27

ADOPTED, 1927.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation E 12; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Absolute Specific Gravity** (of solids and liquids).—The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water<sup>3</sup> at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

Absolute Specific Gravity  $x^{\circ}/x^{\circ}\text{C.}$  .....

where  $x$  is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

Absolute Specific Gravity  $x^{\circ}/y^{\circ}\text{C.}$  .....

where  $x$  is the temperature of the material and  $y$  is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

**Specific Gravity** (of solids and liquids).—The ratio of the weight in air of a given

volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

Specific Gravity  $x^{\circ}/x^{\circ}\text{C.}$  .....

where  $x$  is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

Specific Gravity  $x^{\circ}/y^{\circ}\text{C.}$  .....

where  $x$  is the temperature of the material and  $y$  is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

**Apparent Specific Gravity** (of solids).—The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

Apparent Specific Gravity  $x^{\circ}/x^{\circ}\text{C.}$  .....

<sup>1</sup> Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-8 on Nomenclature and Definitions.

<sup>2</sup> Prior to adoption as standard, these definitions were published as tentative from 1925 to 1927, being revised in 1926 and 1927.

<sup>3</sup> Distilled water boiled vigorously in vacuum.

where  $x$  is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

Apparent Specific Gravity  $x^\circ/y^\circ\text{C.}$  .....

where  $x$  is the temperature of the material and  $y$  is the temperature of the water.

NOTE 1.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by the second definition (specific gravity) would be designated as "apparent specific gravity" and that defined by the first definition (absolute specific gravity) as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

NOTE 2.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

NOTE 3.—In the interest of standardization and simplification, the first form of

statement of specific gravity (a) should be employed wherever practicable.

**Bulk Specific Gravity (of solids).**—The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are *the same*:

Bulk Specific Gravity  $x^\circ/x^\circ\text{C.}$  .....

where  $x$  is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

Bulk Specific Gravity  $x^\circ/y^\circ\text{C.}$  .....

where  $x$  is the temperature of the material and  $y$  is the temperature of the water.

NOTE 1.—See Note 2 under "Apparent Specific Gravity."

NOTE 2.—In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

# Standard Definitions of

## TERMS RELATING TO RHEOLOGICAL PROPERTIES OF MATTER<sup>1</sup>



A.S.T.M. Designation: E 24 - 42

ADOPTED, 1942.<sup>2</sup>

This Standard of the American Society for Testing Materials is issued under the fixed designation E 24; the final number indicates the year of original adoption as standard or, in the case of revision, the year of last revision.

**Consistency.**—That property of a body by virtue of which it tends to resist deformation.

**Plasticity.**—That property of a body by virtue of which it tends to retain its deformation after reduction of the deforming stress to its yield stress.

**Elasticity.**—That property of a body by virtue of which it tends to recover its original size and shape after deformation.

**Liquid.**—A substance which undergoes continuous deformation when subjected to shearing stress.

**Simple Liquid.**—A liquid in which the rate of shear is proportional to the shearing stress. The constant ratio of shearing

stress to rate of shear of a simple liquid is the *viscosity* of the liquid.

**Complex Liquid.**—A liquid in which the rate of shear is not proportional to the shearing stress.

**Solid.**—A substance which undergoes permanent deformation only when subjected to shearing stress in excess of some finite value characteristic of the substance (yield stress).

**Plastic Solid.**—A substance which does not deform under a shearing stress until the stress attains the yield stress, when the solid deforms permanently.

**Elastic Solid.**—A substance in which, for all values of the shearing stress below the rupture stress (shear strength), the strain is fully determined by the stress regardless of whether the stress is increasing or decreasing.

<sup>1</sup> Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Prior to adoption as standard, these definitions were published as tentative from 1934 to 1942, being revised in 1937 and 1940.

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A.S.T.M. TENTATIVES  
RELATING TO NONMETALLIC  
MATERIALS—ELECTRICAL INSULATION,  
PLASTICS, RUBBER, PAPER,  
SHIPPING CONTAINERS, ADHESIVES

*As of December, 1946*

(For revisions or change in status of any of these tentatives,  
see supplementary publications to be issued in 1947 and 1948.)

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*Tentative Specifications and Methods of Test* represent the latest thoughts and practices, and are published by the Society on the recommendations of the committees concerned, prior to adoption as standard.

New and revised Tentatives, issued in 1947 and 1948, will appear in the 1947 and 1948 Supplements to the Book of A.S.T.M. Standards. It is important that these Supplements be consulted in case any change of status has occurred with respect to any of these Tentatives.

Criticisms of these Tentatives should be directed to the Headquarters of the Society, 1916 Race St., Philadelphia 3, Pa.

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In the serial designations prefixed to the following titles, the initial letter, indicative of the general classification, and the first number are permanent. The number following the dash indicates the year of original issue or, in the case of revision, the year of last revision. Thus, tentatives issued or revised during the year 1946 have as their final number, 46. The letter T is appended to the designation of all tentatives.

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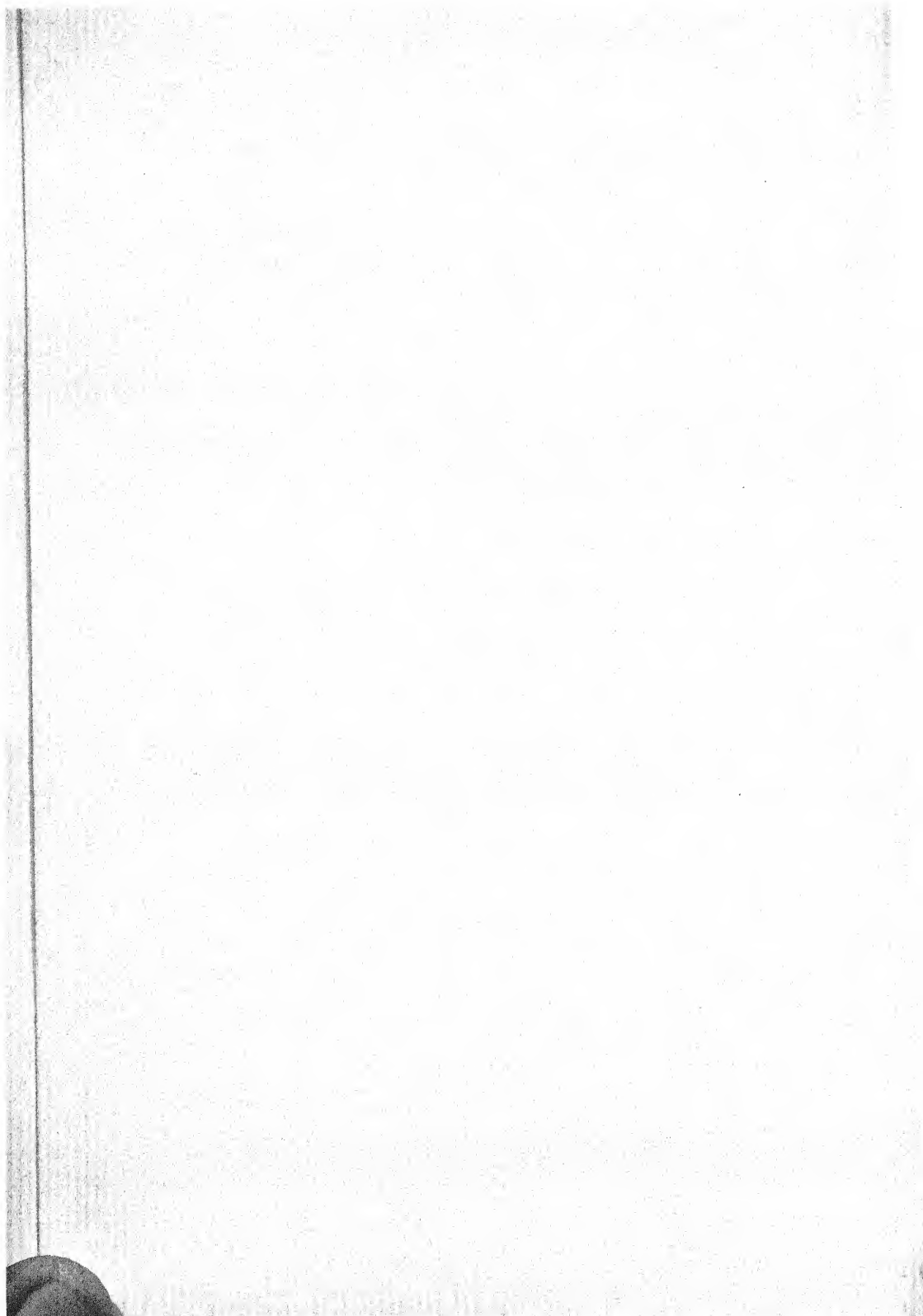
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# Tentative Specifications for

## ORANGE SHELLAC AND OTHER INDIAN LACS FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 784 - 44 T

ISSUED, 1944<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover five grades of orange shellac and other Indian lacs suitable for use as electrical insulation. The grades of lac covered are the customarily manufactured products derived entirely from sticklac (the secretion of *Laccifer Lacca Kerr*) designated grades A, B, C, D, and E. Sticklac

NOTE.—Grades A, B, and C are the commercial rosin-free grades of orange shellac commonly known by these A.S.T.M. designations.<sup>3</sup> Grade D customarily occurs in the form of circular disks about 3 in. in diameter and  $\frac{1}{4}$  in. in thickness and is known to the trade as pure button lac. Grade E is dark garnet in color and is known to the trade as pure garnet lac. Garnet lac also is manufactured in admixture with rosin but this grade is not covered by these specifications.

TABLE I.—REQUIREMENTS FOR ORANGE SHELLAC AND OTHER INDIAN LACS.

	Grade A	Grade B	Grade C	Grade D	Grade E
Matter insoluble in specified hot solvents, max., per cent.....	1.25	2.00	3.00	1.00	0.50
Rosin.....	none	none	none	none	none
Moisture, max., per cent.....	2.0	2.0	2.0	2.0	2.0
Wax, max., per cent.....	5.5	5.5	5.5	4.0	3.5
Orpiment, max., per cent.....	0.025	0.025	0.20	0.025	0.025
Matter soluble in water, max., per cent....	0.5	0.5	0.5	0.5	0.5
Ash, max., per cent.....	1.0	1.0	1.0	0.5	0.5
Polymerization time.....	when specified, shall be within the maximum-minimum range agreed upon by the purchaser and the seller. <sup>4</sup>				
Flow test.....					

<sup>a</sup> Attention is called to the fact that the purchaser and the seller must also agree upon the test temperature and in the case of the flow test upon one of the two methods for determining flow appearing in A.S.T.M. Methods D 411.<sup>4</sup>

and seedlac are not included under these grades.

### Properties

2. The lac covered by these specifications shall conform to the requirements prescribed in Table I.

### Methods of Testing

3. The material shall be sampled and the properties enumerated in these specifications shall be determined in accordance with the Standard Methods of Testing Shellac Used for Electrical Insulation (A.S.T.M. Designation: D 411).<sup>4</sup>

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> See the Tentative Specifications for Orange Shellac and Other Lacs (A.S.T.M. Designation: D 237), 1946 Book of A.S.T.M. Standards, Part II.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



# *Tentative Specifications for*

## FLEXIBLE TREATED COTTON AND RAYON SLEEVING USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 372 - 45 T

ISSUED, 1933; REVISED, 1935, 1937, 1940, 1944, 1945.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover flexible treated cotton and rayon sleeving in five different grades, as follows:

*Grade A-1.*—A cotton or rayon braided sleeving impregnated and coated with a continuous film of an oil varnish. This treated sleeving shall conform to the requirements of these specifications, including the heat-endurance test (Section 6). It is suitable for applications involving high-temperature baking or continuous exposure to elevated temperatures, and for apparatus into which hot potting compounds are poured for sealing purposes.

*Grade A-2.*—A cotton or rayon braided sleeving coated with a continuous film of a lacquer. In some sizes the sleeving may be impregnated with a suitable medium before being coated. This treated sleeving shall conform to the requirements of these specifications, except the heat-endurance test (Section 6).

It is generally suitable for applications not involving exposure to high temperature.

*Grade B-1.*—A treated sleeving having a construction similar to grade A-1. It shall conform to the requirements of these specifications, including the heat-endurance test (Section 6). This grade of treated sleeving has intermediate dielectric strength and resistance to aging.

*Grade B-2.*—A treated sleeving having a construction similar to grade A-2. It shall conform to the requirements of these specifications, except the heat-endurance test (Section 6). This grade of treated sleeving has intermediate dielectric strength and resistance to aging.

*Grade C.*—A braided cotton or rayon sleeving impregnated with an oil varnish. It shall have maximum flexibility. It is generally known as "saturated sleeving," and has a lower dielectric strength than grades B-1 or B-2.

NOTE 1.—*Oil Varnish* is defined<sup>3</sup> as a varnish which contains resin and drying oil as the basic film-forming ingredients and is converted to a solid film primarily by chemical reaction.

<sup>3</sup> This definition appears in the Standard Definitions of Terms Relating to Paint, Varnish, Lacquer, and Related Products (A.S.T.M. Designation: D 16), 1946 Book of A.S.T.M. Standards, Part II.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, June 27, 1945.

NOTE 2.—For the purpose of these specifications, lacquer is defined as a spirit varnish, containing synthetic resins as the principal film-forming ingredients, which is converted to a solid film primarily by solvent evaporation.

### Material

2. (a) The base material of the treated sleeving shall be good quality cotton or rayon braided sleeving.

(b) The cotton or rayon sleeving shall be uniformly treated, and the finished treated sleeving shall be reasonably free from defects such as pimples, blisters, wrinkles, cracks, and similar imperfections.

### Color

3. Yellow, black, and red shall be considered standard colors. Other colors shall be considered special. The color desired shall be specified in the purchase order.

### Dielectric Strength

4. (a) Grades A-1 and A-2 treated sleeving shall show an average value in the short-time dielectric strength test of not less than 7000 v., and no one of the ten puncturing voltages shall be less than 5000 v.

(b) Grades B-1 and B-2 treated sleeving shall show an average value in the short-time dielectric strength test of not less than 4000 v., and no one of the ten puncturing voltages shall be less than 2500 v.

(c) Grade C treated sleeving shall show an average value in the short-time dielectric strength test of not less than 1200 v., and no one of the ten puncturing voltages shall be less than 800 v.

### Aging Test

5. The number of hours of baking in the aging test during which no cracking of the tubing occurs when bent through 180 deg. over the rod specified in Section 14 of the Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350)<sup>4</sup> shall be not less than the following:

Grades A-1, A-2, and C..... 96 hr.  
Grades B-1 and B-2..... 72 hr.

### Heat Endurance

6. When tested for heat endurance in accordance with Section 17 of the A.S.T.M. Standard Methods D 350, grades A-1, B-1, and C treated sleeving shall neither soften, blister, nor flow.

### Rate of Burning

7. All grades of treated sleeving furnished under these specifications shall require at least 45 sec. to burn a length of 1 in. (2.54 cm.).

TABLE 1.—A.S.T.M. STANDARD SIZES FOR FLEXIBLE TREATED SLEEVING.

Size	Inside Diameter, in.		
	Maximum	Minimum	Nominal
1-in.....	1.036	1.000	1.018
1 1/8-in.....	0.911	0.875	0.893
1 1/4-in.....	0.786	0.750	0.768
1 3/8-in.....	0.655	0.625	0.640
1 1/2-in.....	0.524	0.500	0.512
1 5/8-in.....	0.462	0.438	0.450
1 3/4-in.....	0.399	0.375	0.387
No. 0.....	0.347	0.325	0.330
No. 1.....	0.311	0.289	0.294
No. 2.....	0.278	0.258	0.263
No. 3.....	0.249	0.229	0.234
No. 4.....	0.224	0.204	0.208
No. 5.....	0.198	0.182	0.186
No. 6.....	0.178	0.162	0.166
No. 7.....	0.158	0.144	0.148
No. 8.....	0.141	0.129	0.133
No. 9.....	0.124	0.114	0.118
No. 10.....	0.112	0.102	0.106
No. 11.....	0.101	0.091	0.095
No. 12.....	0.089	0.081	0.085
No. 13.....	0.080	0.072	0.076
No. 14.....	0.072	0.064	0.066
No. 15.....	0.067	0.057	0.059
No. 16.....	0.061	0.051	0.053
No. 17.....	0.054	0.045	0.047
No. 18.....	0.049	0.040	0.042
No. 20.....	0.039	0.032	0.034
No. 24.....	0.027	0.	0.022

NOTE 1.—The minimum inside diameters prescribed in this table for sizes Nos. 0 to 24, inclusive, are the same as the diameters of copper wire for corresponding A. w. g. or B. & S. gage sizes.

NOTE 2.—Sizes Nos. 15 and 17 are special and may eventually be eliminated.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

**Dimensions**

8. (a) *Inside Diameters*.—The inside diameters of flexible treated sleeving shall conform to the requirements prescribed in Table I.

(b) *Commercial Lengths*.—Unless otherwise specified, treated cotton and rayon sleeving in all sizes shall be supplied straight in a nominal length of 36 in. When so specified on individual orders treated sleeving in sizes Nos. 24 to 7, inclusive, may be supplied in continuous lengths.

**Methods of Testing**

9. Flexible treated sleeving shall be sampled, conditioned, and tested in accordance with the Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (A.S.T.M. Designation: D 350) of the American Society for Testing Materials.<sup>4</sup>

**Packing**

10. Unless otherwise specified by the purchaser, the treated sleeving shall be packed straight in packages conforming to usual trade customs.

**Marking**

11. Each package shall be marked

with the name or trade mark of the manufacturer, the total number of feet, and the size, color, and grade of the treated sleeving.

**Inspection**

12. The treated sleeving shall be inspected and tested within two weeks of the date of receipt by the purchaser.

**Rejection**

13. If the results of any test do not conform to the requirements prescribed in these specifications for the grade specified, that test shall be repeated on two additional specimens from the same lot, each of which shall conform to the requirements specified. If either of these two additional specimens fails, the lot of treated sleeving represented by that specimen may be rejected at the option of the purchaser.

**Rehearing**

14. Samples of rejected material shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of test, the manufacturer may make claim for a rehearing within that time.

# *Tentative Specifications for*

## BLACK BIAS-CUT VARNISHED CLOTH TAPE USED FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 373 - 44 T

ISSUED, 1933; REVISED, 1934, 1936, 1937, 1939, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover black seamless bias-cut varnished cloth tapes 7 to 12 mils in thickness to be used for insulating bus bars, joints and terminals of electric wires and cables, and other electrical apparatus connections.

NOTE.—Orders for material under these specifications shall include the following information:

The desired nominal thickness, width, and length (Section 10),

Type of surface, greasy or tacky (Section 3),

Type of packing, dry or oil (Section 12),

Number of rolls in package (Section 12 (b) and (c)), and

Marking on package (Section 13).

### Manufacture

2. (a) *Materials*.—The cloth and the number of coats of varnish applied shall be such as to produce a product which will meet the requirements of these specifications.

(b) *Splices*.—Splices are those joints made by the manufacturer of the un-

treated cloth. Splices shall not occur more than once in each roll, except that rolls having a length of 6 yd. or less shall contain no splices.

(c) *Defects*.—In general, the tape shall be free from wrinkles, excessive pebbling, blisters, and other imperfections.

### Surface

3. Tapes are roughly divisible into two classes: "greasy" and "tacky." Greasy tapes shall have the property of sliding freely, one layer upon another, when moved easily between two fingers.

### Breaking Strength

4. (a) The breaking strength requirement varies with the nominal thickness of the tape. Let  $t$  represent the nominal thickness in mils; then, the average breaking strength of the specimens shall be not less than  $35 + (t - 7)$  lb. per inch of width. If the first specimen from a roll does not conform to this requirement, tests of two additional specimens from the same roll shall be made and the average of these three tests shall conform to the above requirement. The

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, December 22, 1944.

breaking strength of any of the specimens tested shall be not less than  $29 + (t - 7)$  lb. per inch of width.

(b) The breaking strength of the "jointed selvages" shall be not less than 80 per cent of the values specified in Paragraph (a).

### Elongation

5. For tapes of nominal width not less than 0.5 in., the elongation of one specimen from each of the selected rolls shall be measured at a temperature  $T$  between 20 and 30 C. and shall be not less than that specified in the following table:

Nominal Thickness	Load, lb. per inch of Tape Width	Elongation, per cent
7 mils.....	5	$1.7 + (T - 20) \times 0.1$
Over 7 mils.....	6	$1.7 + (T - 20) \times 0.1$

### Resistance to Oil

6. (a) One specimen from each of the selected rolls shall be subjected to test for resistance to oil as described in the methods referred to in Section 11. The oil used shall be an insulating oil having a flash point of not less than 135 C. (275 F.), as determined by the Cleveland open cup in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92) of the American Society for Testing Materials.<sup>3</sup> The varnished film shall show no evidence of disintegration in the oil nor flake either in the oil or on the blotter used in the test.

(b) The specimens tested for resistance to oil shall be used for electrical tests as described in Section 9.

### Dielectric Strength, as Received

7. (a) Ten punctures shall be made on one specimen from each roll as received. The dielectric strength values

are the puncture values in volts divided by the average of the ten thickness readings on the specimens in mils. The average of the ten dielectric strength values for each roll as received, shall conform to the requirements prescribed in Table I. The dielectric strength values when analyzed to determine the uniformity of the material shall conform to the requirements specified in Paragraphs (b) and (c).

TABLE I.—DIELECTRIC STRENGTH REQUIREMENTS FOR VARNISHED CLOTH TAPE.

Nominal Thickness	Dielectric Strength, minimum average volts per mil		
	As Received	Under 6 per cent Elongation	After Hot Oil
Over 10 mils.....	1100	1000	1100
9 and 10 mils, incl.....	1000	900	1000
Under 9 mils.....	900	800	900

(b) *Uniformity Within Roll.*—The deviation factor for each roll in each shipment shall be not more than 0.08 when calculated as follows:

$$\text{Deviation factor of roll} = \frac{h - l}{h + l}$$

where:

$h$  = average of the five highest dielectric strength values of the roll in volts per mil, and

$l$  = average of the five lowest dielectric strength values of the roll in volts per mil.

(c) *Deviation of Each Roll of Shipment from Average.*—The deviation factor for each roll in each shipment shall be not more than 0.07 when calculated as follows:

$$\text{Deviation of each roll of shipment from average} = \frac{a - l}{a + l}$$

where:

$a$  = average dielectric strength of the entire shipment in volts per mil, and

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

$l$  = average of the five lowest dielectric strength values of the roll in volts per mil.

#### Dielectric Strength, Under Elongation

8. Five punctures shall be made inside the gage length on each specimen while elongated to the prescribed value. The average of the five dielectric strength values for each roll shall conform to the requirements prescribed in Table I.

NOTE.—In the use of tape it is probable that it may be elongated more than 6 per cent. In order to obtain dielectric strength data at a higher percentage elongation, it is recommended that tests be made at 12 per cent elongation in addition to the specified 6 per cent elongation.

#### Dielectric Strength, After Hot Oil

9. Five punctures shall be made on each specimen after the test for resistance to oil specified in Section 6. The average of the five dielectric strength values for each roll shall conform to the requirements prescribed in Table I.

#### Dimensions and Permissible Variations

10. (a) *Thickness*.—The nominal thickness shall be specified in the purchase order. The average thickness shall be determined from ten measurements taken on a specimen before testing in accordance with Section 7. The average thickness shall not vary more than plus or minus 0.001 in. from the specified nominal thickness, and no individual measurement shall vary more than plus 0.002 or minus 0.001 in. for tapes from 5 to 9 mils in thickness, nor more than plus or minus 0.003 in. for tapes 10 to 12 mils in thickness, from the specified nominal thickness.

(b) *Width*.—The nominal width shall be specified in the purchase order. Individual measurements on all the samples selected under Section 11 shall not vary more than plus or minus  $\frac{1}{32}$  in. from the specified nominal width for tapes 1 in. or less in width, nor more

than plus or minus  $\frac{1}{16}$  in. for tapes over 1 in. in width.

(c) *Length*.—The nominal length or yards per roll shall be specified in the purchase order. The measured lengths of individual rolls selected under Section 11 shall be not less than that specified.

#### Methods of Testing

11. (a) Varnished cloth tape shall be sampled, conditioned, and tested in accordance with the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295) of the American Society for Testing Materials.<sup>4</sup>

(b) All specimens taken from a single roll shall be numbered or marked so that failures can be identified with the roll.

#### Packing

12. (a) The purchaser shall specify whether tape is to be supplied dry or immersed in oil.

(b) *Dry Packing*.—In the case of dry tape, the purchaser shall specify the number of rolls per package and whether each roll shall be dipped or whether each package shall be dipped in a moisture-resistant wax or neither or both. The wrapping shall be secure and shall protect the contents.

(c) *Packing in Oil*.—Oil-immersed tape shall be supplied in suitable oiltight cans with easily removable lids. The purchaser shall specify the number, width, and length of rolls to be supplied in each can. The type of oil used for packing shall be subject to the approval of the purchaser.

#### Marking

13. The purchaser shall specify the marking desired on each package or

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

can; unless the marking is definitely specified, the package shall conform with the usual trade customs.

### Inspection and Testing

14. The material shall be inspected and tested within three weeks of its receipt by the purchaser.

### Rejection

15. (a) The purchaser reserves the right to reject any part of the shipment that does not conform to the requirements for packing and marking as specified in Sections 12 and 13.

(b) The failure of a sample roll to conform to the requirements of one or more of the sections enumerated under Paragraph (c) shall be counted as only one roll failure.

(c) The failure of 20 per cent or more of the sample rolls to conform to the requirements of any one of Sections 2 (b) and (c), and 3, 4, 5, 6, 7, 8, 9, and 10, or failure of 30 per cent or more of the sample rolls to meet the requirements of

any of these sections shall constitute cause for rejection of the entire shipment without further tests. (See Explanatory Note.)

### Claims

16. Rejection claims shall be made to the seller in writing within four weeks of receipt of the material by the purchaser. The results of tests made by the purchaser on which rejection is based shall be furnished to the seller, who shall, within one week of receipt of all such claims, either agree to satisfy the claim or send a representative to the plant of the purchaser to resample the shipment. Samples so selected shall be sealed and submitted to an umpire, mutually agreeable to the purchaser and seller, whose decision should be final.

### Settlement of Claims

17. The expense of the umpire tests shall be paid by the loser. In the case of rejection being established, the seller shall pay the freight both ways on the rejected material.

### EXPLANATORY NOTE

NOTE.—The following illustrates a convenient method of recording failures of sampled rolls:

Roll No.	Failures Under Sections									Roll Failures
	2(b), 2(c)	3	4	5	6	7	8	9	10(a), 10(b), 10(c)	
1.....										
2.....			x							1
3.....								x		1
4.....										
5.....										
6.....										
7.....			x							1
8.....										
9.....										
10.....										
Total.....			2 <sup>a</sup>					1	1	3 <sup>b</sup>

<sup>a</sup> Two failures (equal to 20 per cent of rolls sampled) under a single section causes rejection under the 20 per cent clause.

<sup>b</sup> Three roll failures (equal to 30 per cent of rolls sampled) causes rejection under the 30 per cent clause.



# Tentative Methods of TESTING VARNISHES USED FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 115 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods cover tests for varnishes which are applied by brushing, dipping, or spraying and intended primarily for the purpose of providing electrical insulation. The tests appear in the following order:

Density .....	3
Viscosity .....	4 to 6
Flash Point .....	7
Time of Drying .....	8
Dielectric Strength of Varnish in the Solid State:	
Method A .....	9 to 13
Method B .....	14 to 16
Heat Endurance (Bend Test) .....	17 and 18
Oil Proof Test (Oil Resistance) .....	19
Draining Test (Working Viscosity) ..	20 and 21
Nonvolatile Matter by Weight .....	22 to 24
Volatile Matter by Volume .....	25 to 28
Dielectric Strength of Varnish in the Liquid State .....	
	29 to 32

## Test Specimens

2. (a) For tests which are to be performed upon the varnish itself, the specimen shall consist of an adequate, representative sample which, until re-

quired, shall be kept at room temperature in a nearly filled, tightly sealed container to avoid skin formation and escape of solvents.

(b) For tests which are to be performed upon the varnish as a film on a copper base, copper strips  $1\frac{1}{4}$  in. in width, 8 in. in length, and  $0.005 \pm 0.0003$  in. in thickness shall be used, unless otherwise specified. The thickness of these strips shall be measured to the nearest 0.0001 in. They shall be cleaned first with a suitable solvent, then polished thoroughly with No. 000 steel wool, and finally wiped free of any fingerprints or metal particles with the solvent and a lint-free cloth. If the strips are not to be used immediately, they shall be kept stored in a noncorrosive varnish solvent (Note 1).

NOTE 1.—Toluol- or xylol-alcohol (1:1) have been found suitable cleaning solvents, and V.M. & P. naphtha a suitable solvent in which to store the strips.

(c) Test specimens shall be prepared after the strips have been wiped clean and dry by dipping them into a tank of the varnish which has been adjusted to a proper consistency and allowed to stand

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946. Prior to their present publication as tentative, these methods were published as tentative from 1921 to 1941, being revised in 1923, 1924, 1925, 1926, 1928, 1929, 1930, 1931, 1934, 1935, 1936, 1938, and 1939. They were adopted in 1941 and published as standard from 1941 to 1946. The methods were revised and republished as tentative in 1946.



covered until free of air bubbles (not to exceed 1 hr.). The proper consistency of the varnish shall be found by trial. It shall be such that when the strips are dipped into the varnish at a temperature of approximately 25 C. (77 F.) and are withdrawn slowly and uniformly at a rate to be agreed upon by buyer and seller the average thickness of the film remaining on each side of a strip when dry shall be between 0.0009 and 0.0010 in. (Note 2). In the absence of any special agreement a withdrawal rate of 4 in. per min. is suggested. The average thickness shall be calculated by averaging at least six measurements taken along the length of the strip and over  $\frac{1}{8}$  in. from either edge. Thickness measurements shall be made in accordance with method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374).<sup>3</sup>

NOTE 2.—It is recognized that the thickness cannot be measured with the precision stated, but a close control of the thickness of the varnish film is desired. With the method specified, the actual average thickness should be within plus or minus 0.0002 in. of the measured thickness.

(d) Except where time of drying is the property being measured, specimens following each dip shall be suspended vertically in dipping position and dried in dust-free air for such times and at such temperatures as the buyer and the seller agree are suitable. With baking varnishes it may be desirable to allow the specimens to drain at approximately 25 C. (77 F.) until the varnish is set, so that an impression left by pressing lightly with the finger about 2 in. from the bottom edge will remain. When specimens are to be given two dips the consistency of the varnish shall be readjusted if necessary before the second dip, and it shall be made in the reverse direction to the first.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## DENSITY

### Procedure

3. The density of the varnish shall be determined in grams per cubic centimeter at  $25 \pm 1$  C. and recorded to the nearest 0.001 g. per cu. cm. using a wide-mouthed 25-ml. pycnometer of the Hubbard-Carmick type (Note). The volume of the pycnometer shall be obtained by weighing the pycnometer first empty and then filled with distilled water at 25 C., (77 F.) and dividing the difference in weight by 0.997. The density shall be determined by dividing the weight of varnish by its volume. For convenience and accuracy a curve shall be plotted for each pycnometer showing relation between weight of pycnometer plus varnish and the density.

NOTE.—The Hubbard-Carmick type of pycnometer permits the escape of air bubbles from the more viscous types of varnishes.

## VISCOSITY

### Apparatus

4. Viscosimeters used in this test shall be kept calibrated over the range of viscosity of the varnishes to be tested by means of oils verified as to absolute viscosity at 25 C. by the National Bureau of Standards. A calibration curve showing the relation between viscosity in absolute units and the instrument readings shall be used.

NOTE.—A description of the MacMichael viscosimeter as remodeled and improved for varnish testing, together with directions for its calibration in absolute units, appears in the Appendix.

### Procedure

5. Sufficient varnish for a test (Section 2 (a)) shall be brought to a uniform temperature of  $25 \pm 0.2$  C. and poured into the viscosimeter which shall have been adjusted to, and shall be maintained at, this temperature. The test shall be

made immediately to avoid evaporation, or formation of skin on the surface of the varnish. Results shall be stated in terms of absolute viscosity, in centipoises (Note). Two specimens shall be tested and the mean value reported, provided the average deviation of a single observation from the mean is not greater than 2 per cent. Otherwise, the instrument and method used shall be checked, and additional tests made until the average deviation from the mean does not exceed 2 per cent.

NOTE.—The absolute viscosity of a material or its viscosity in absolute units (c.g.s., poises) has been defined as the force in dynes required to move, at a velocity of 1 cm. per sec., one surface having an area of 1 sq. cm. past another parallel like surface 1 cm. away, overcoming the resistance to shear of the material filling the space between. One centipoise = 0.01 poise.

#### Report

6. The report shall include the following:

(1) Individual viscosity readings in centipoises and corresponding varnish temperatures to the nearest 0.1 C.

(2) Mean value of the viscosity in centipoises,

(3) Average deviation of individual observations from the mean viscosity in centipoises and in percentage of the mean, and

(4) Type of viscosimeter used.

#### FLASH POINT

##### Procedure

7. The flash point shall be determined in accordance with the Standard Method of Test for Flash Point by Means of the Pensky-Martens Closed Tester (A.S.T. M. Designation: D 93).<sup>4</sup>

#### TIME OF DRYING

##### Procedure

8. (a) Specimens shall be dipped once as described in Section 2 (c) and suspended for drying in dust-free air at such

temperature as the purchaser and the seller have agreed upon (Note 1). At the end of the first 30 min. and, again at the end of each 10-min. period thereafter, one specimen shall be taken from the oven and examined. In the case of slow-drying varnishes these periods may be lengthened at the discretion of the operator.

NOTE 1.—Where an oven is used its particular size and ventilation have a considerable effect on the drying time of varnishes. It is preferable that the oven conform with some definite or standard specification such as that given in the Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).<sup>4</sup>

(b) The varnish shall be considered dry (Note 2) when a piece of kraft paper  $1\frac{1}{2}$  in. in width, 6 in. in length, and approximately 0.0025 in. in thickness does not adhere to the varnish when it is pressed on the surface of the varnish for 1 min. by a cylindrical 1-lb. weight, 1 in. in diameter. The kraft paper shall be applied in the vicinity of the center of the specimen and at right angles to it. The test shall be made at a temperature of approximately 25 C. (77 F.).

NOTE 2.—The drying time of varnishes may vary with the base on which the varnish is dried. It should not be expected that varnishes will dry alike on all materials or even on all metals. Certain types of varnishes dry with what is commonly known as "tack," but these types are rare. However, when testing them for drying time, it will be found that the kraft paper adheres to the varnish film and offers a certain amount of resistance to removal. On continued drying, the resistance to removal reaches constancy and is not changed by further drying. Therefore, the drying time is reported as the number of hours required to first reach constancy and the varnish should be reported as drying with a "tack."

#### DIELECTRIC STRENGTH OF DRIED VARNISH FILM

##### Method A

##### Purpose

9. (a) This method determines the

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

suitability of an insulating varnish for use in certain electrical applications by determining its dielectric strength under different conditions of temperature and after immersion in water. Method A provides for the determination of dielectric strength under the conditions listed in Section 12.

(b) Much more information about a varnish can be obtained by making the test under all three conditions listed in Section 12 than by making it under only one condition. The relation which the dielectric strength tests for a given varnish show to one another is of quite as much significance as the values for the different tests considered separately.

### Electrodes

10. Tests shall be made between two metal cylinders, 2 in. in diameter, and with edges rounded to a radius of  $\frac{1}{4}$  in. The pressure between electrodes shall be approximately 1.1 lb.

### Test Specimens

11. Two specimens for dielectric strength tests shall be prepared as described in Section 2, except that copper sheets 8-in. square and 0.005 in. in thickness shall be used. Each specimen shall be dipped twice and the final thickness of the film of varnish on each side of the specimen shall be between 0.0018 and 0.002 in.

### Conditioning

12. The dielectric strength shall be determined under the following conditions:

- (1) At room temperature in air (Note 1),
- (2) At 75 C. in air (Notes 1 and 2), and
- (3) At room temperature on specimens which shall have been immersed in water at a temperature of approximately 25 C. (77 F.) for a period of 24 hr. (Note 3).

NOTE 1.—Dielectric strength determinations may also be made in oil at room temperature and at 75 C. (167 F.). With this exception the

procedure for test is the same as outlined above. If tests are made in oil, this fact shall be definitely reported with the results.

NOTE 2.—The tests at 75 C. (167 F.) shall be made in air in an analytical type of electrically heated oven. The electrodes shall be at oven temperature before the test is started. Tests should be started 15 min. after the specimen has been introduced into the oven and placed between the electrodes, and completed before 15 min. additional have elapsed.

NOTE 3.—In testing specimens after immersion in water, only one specimen should be removed from the water at a time. The specimen after removal from the water should be carefully dried by placing between sheets of dry blotting paper and pressing down gently with the hand over the entire surface. If necessary, the blotting procedure should be repeated until the specimen is free from surface moisture. The specimen should then be tested immediately. If specimens are allowed to remain out of water for even a short time before testing, they dry out to a greater or less extent and the test is likely to be misleading.

### Procedure

13. (a) The dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149)<sup>5</sup> using the short-time test and increasing the voltage at the rate of 0.5 kv. per sec.

(b) Ten punctures shall be made, five on each of two specimens.

(c) After testing, the thickness of the specimen shall be determined by making five measurements on a strip cut diagonally across the specimen. The measurements shall be spaced approximately equally along the strip. The average of the five measurements, less the thickness of the copper, shall be used for calculating the dielectric strength in volts per mil.

### Method B<sup>5</sup>

### Test Specimens

14. (a) Specimens for the dielectric strength test shall be made from pieces

<sup>5</sup> This method is that described in Bureau of Ships Ad Interim Specification 52V13 (INT) for Varnish, Insulating (Electrical), November 1, 1943.

of smooth sheet copper approximately 8 in. in length, 3.5 in. in width, and 0.005 in. in thickness, thoroughly cleaned with xylol-alcohol solvent (1:1) and rubbed dry with clean cheesecloth. Two sheets shall be placed together and sealed at the edges so that the varnish film is obtained on one side of each copper sheet only. The assembly shall be dipped twice in the varnish to be tested, once in each direction in order to give a more uniform thickness of coating. The dipping shall be done at room temperature of approximately 25 C. (77 F.) and 50 per cent relative humidity. The consistency of the varnish shall first have been so adjusted by trial that, when dry, the final thickness of the film of varnish on one side of the specimen shall be not less than 0.0018 nor more than 0.002 in. The varnish shall have been allowed to stand until free of air bubbles.

(b) Specimens of air-drying varnish shall be dried in free air after each dip in the same vertical position in which they were dipped, at approximately 25 C. (77 F.) and 50 per cent relative humidity for a period of 24 hrs. Specimens of baking varnishes shall be baked after each dip in the same vertical position in which they were dipped, at 100 C. (212 F.) for 16 hrs., or at such temperature and for such time as specified by the manufacturer.

### Conditioning

15. Specimens shall be tested without further treatment following drying or baking, and also after a 24-hr. immersion in distilled water at approximately 25 C. (77 F.). Specimens of baking varnish shall be allowed to cool 15 min. before immersion. Upon removal from the water, the surface water shall be wiped off and dielectric strength tests made immediately.

### Procedure

16. (a) The specimens which were fastened together for dipping and drying

or baking shall be separated without bending, and the dielectric strength of each film shall be determined separately. Using electrodes as described in Section 10, an alternating test potential shall be applied at a low value and gradually raised at a rate such that puncture will occur in approximately 10 to 15 secs. The frequency of the test potential shall not be greater than 100 cycles per sec., and each part of the testing apparatus shall have a continuous rating of not less than 2 kva. (preferably larger). The wave form shall be a sine curve, and the voltage shall be measured by an approved method. The voltage may be controlled by any approved method which does not distort the wave more than 10 per cent from a sinusoidal shape and which does not subdivide the voltage in steps greater than 500 v.

(b) Ten punctures shall be made at various points on the specimens.

(c) Ten thickness measurements shall be made with a dial-type micrometer on each copper panel and at the same points on the coated panels. The difference in averages of these two sets of measurements shall be used as the thickness of the varnish film on each panel in calculating dielectric strength in volts per mil. The breakdown voltage shall be reported for each of the 10 tests, together with the average volts per mil and the average film thickness.

### HEAT ENDURANCE TEST

#### Procedure

17. (a) Test specimens, which have been dipped and dried twice as described in Section 2 (b) to (d) to give a coating 0.0017 to 0.0021 in. in thickness on each side of the copper (Note 1), shall be placed in a uniformly heated oven (Notes 2 and 3) in which the temperature shall be maintained at 105 to 110 C. (221 to 230 F.) (Note 4). A specimen shall be removed at the end of the first 24 hr., and every 24 hr. thereafter.

NOTE 1.—As the “grain” of the copper influences the result of the test, the specimens for the heat endurance test should be prepared by cutting the copper strips parallel with the direction of rolling.

NOTE 2.—The size oven and the amount of ventilation have a considerable effect on the heat endurance of varnish films. For this reason the analytical type of electrically-heated oven should be used, and if different ovens are used they should be checked with control samples in order to determine whether they will yield comparable results.

NOTE 3.—Because of temperature variations between different levels in the oven, the specimen should be placed in the oven with the 8-in. dimension horizontal and the 1½-in. dimension vertical. All specimens should be at the same level in the oven.

NOTE 4.—The temperature of the oven should be held as closely as possible to the mean (107.5 C., 225.5 F.). A difference of 5 C. (9 F.) in this temperature range, when continuously maintained, has a very considerable effect (approximately 25 per cent) on the life test of a varnish.

(b) Each specimen, after heating, shall be tested at a temperature of approximately 25 C. (77 F.) by bending it through 180 deg. around a rod 0.125 in. in diameter.

### Report

18. The number of hours of baking of the specimen which first showed cracking of the varnish film when bent around the rod shall be reported, together with the make and type number of the oven used.

## OIL RESISTANCE

### Procedure

19. Two inches shall be cut away from one end of test specimens which have been dipped and dried twice as described in Section 2 (b), (c), and (d). Four inches of the cut ends of the specimens shall be immersed for 48 hr. in transformer oil at 105 to 110 C. (221 to 230 F.). A container of oil like that in which the specimens have been immersed shall be heated similarly. The transformer oil shall conform with the require-

ments given in Table I. The specimens shall be wiped with a dry, clean, white cloth and the specimen, the oil, and the cloth shall be examined to determine whether or not the varnish has been affected. The following shall be inter-

TABLE I. TRANSFORMER OIL REQUIREMENTS.<sup>a</sup>

Aniline point, deg. ....	150 to 170
Color. ....	1
Flash point, min. deg. Cent. ....	130
Free and corrosive sulfur. ....	none
Neutralization number, max. ....	0.02
Specific gravity, 15.56/15.56 C. ....	0.885 to 0.900
Viscosity, Saybolt Universal at 100 F., sec. ....	50 to 60

<sup>a</sup> Aniline point shall be determined by the Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611)<sup>4</sup> and all other values by Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117)<sup>5</sup>. In the latter method neutralization number shall be determined by the Tentative Method of Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration. (A.S.T.M. Designation: D 663).<sup>4</sup>

preted and reported as evidence of attack:

(1) Soiling of the cloth, which appears due to softened or disintegrated varnish,

(2) Blistering, wrinkling, disintegration, or separation of the varnish films, and

(3) Discoloration produced by the specimens in the containing oil (Note).

NOTE.—Discoloration produced by heat alone (which should be slight in a satisfactory test oil) shall be discounted by comparison with the “blank.” With black varnishes, slight bleeding of color into the oil is not to be regarded as evidence of failure, if there is no other evidence of attack.

## DRAINING TEST

(Also known as “Working Viscosity” Test)

### Procedure

20. A strip of sheet copper or brass 1½ in. in width, 14 in. in length, and 0.005 ± 0.0003 in. in thickness shall be immersed in the varnish (see Section 2 (a))

at approximately 25 C. (77 F.) up to a line previously drawn across the strip 1 in. from the top. The varnish shall be free of air bubbles. The strip shall be withdrawn at a slow and uniform rate (about 15 in. per min.) and shall be allowed to drain thoroughly at room temperature while suspended vertically. It shall then be dried as described in Section 2 (d) and its thickness measured at points 2, 7, and 12 in., respectively, from the line to which the specimen was immersed.

### Report

21. The thickness of each film in mils at the three points specified in Section 20 shall be recorded. The difference between the thickness at the upper point (2 in.) and that at the lower point (12 in.) shall be taken as a measure of the variation in the film thickness caused by draining.

### NONVOLATILE MATTER BY WEIGHT

#### Apparatus

22. The apparatus shall consist of the following:

(a) *Chemical Balance.*

(b) *Thermometer.*—A glass thermometer having a range of 0 to 150 or 200 C. and accurate within 1 C.

(c) *Oven.*—A constant-temperature oven.

(d) *Bottle and Drying Dishes.*—A stoppered bottle and flat-bottom metal drying dishes. Each metal drying dish shall have an inside diameter of approximately  $2\frac{3}{4}$  in. and a depth of approximately  $\frac{5}{16}$  in.

NOTE.—A standard single-friction tin can cover having a diameter of  $2\frac{1}{8}$  in. is suitable for use as a drying dish.

### Procedure

23. (a) *Preparing Specimens.*—A portion of the sample of varnish (Section 2

(a)) shall be placed in a stoppered bottle or weighing pipette and weighed. A specimen weighing approximately 1.5 g. shall be transferred from the weighed stoppered bottle to a weighed drying dish which has previously been heated for 30 min. at 135 C. and cooled in a desiccator. The stoppered bottle with the remaining contents shall be weighed again. The exact weight of the specimen transferred to the drying dish shall be determined by difference. A total of three specimens shall be prepared from the contents of the stoppered bottle.

(b) *Drying Specimens.*—The specimens shall be placed in the constant-temperature oven within 30 min. after preparation. The specimens shall be heated for a period of 3 hr. at the temperature designated, as follows:

Impregnating varnishes of the phenolic-resin type.....	132 to 138 C.
Other types of insulating varnishes.....	105 to 110 C.

NOTE.—In a gravity type of oven which depends upon the natural circulation of air for uniformity of temperature, only one shelf must be used for supporting the specimens, and the bulb of the thermometer for indicating the temperature shall be in close proximity to the specimens.

(c) *Weighing Dried Specimens.*—At the termination of the 3-hr. heating period, the specimens shall be removed to a desiccator and cooled to room temperature. Each specimen shall be weighed immediately upon removal from the desiccator.

### Calculation and Report

24. (a) The percentage of nonvolatile matter by weight shall be calculated as the ratio of the weight of the dried specimen to the weight of the specimen in the original state expressed as a percentage.

(b) The report shall include the following:

(1) Type of varnish tested,



- (2) Oven temperature, and
- (3) Percentage of nonvolatile matter.

### VOLATILE MATTER BY VOLUME

#### Purpose

25. The purpose of this test is to determine the volume percentage of volatile matter (solvent) in a varnish, and to afford a means of arriving at the volume composition of the varnish with respect to volatile (solvent) and nonvolatile (base) constituents.

#### Procedure

26. (a) About 100 ml. of varnish (Section 2 (a)) shall be distilled in accordance with the Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86).<sup>4</sup> The distillation shall be "qualitative" instead of "quantitative" as given in Section 4 of the Standard Method D 86; that is, it is not necessary to record the volume of distillate recovered or the temperature of the initial boiling point or end point.

(b) The distillation shall be continued until a suitable end point indicates that all the volatile matter (solvent) has been distilled off.

NOTE.—In the distillation of varnishes the end point differs with the various types and compositions. In all cases, however, the rate of distillation slows down as the end point is approached and, when the end point is reached, acrolein (formed in the decomposition of the varnish base) is evolved and may be detected by its characteristic odor. In addition to the evolution of acrolein, the following phenomena are suitable indicators of end point:

(1) When the end point of the solvent is considerably lower than the initial boiling point of the base, the end point of the distillation is indicated by the temperature reaching a maximum and then starting to fall consistently, or

(2) When the end point of the solvent and the initial boiling point of the base are too close, or overlap, the above temperature drop does not occur. The end point of the distillation is then indicated by a sudden foaming up of the residual base in the distillation flask; or, if the distillation has been accompanied by foaming,

the end point is indicated by a breaking of surface tension with a consequent cessation of foaming.

(c) *Density*.—The density of the volatile matter (solvent) recovered by distillation shall be determined in accordance with the procedure described in Section 3.

#### Calculation

27. (a) *Volatile Matter by Weight*.—The difference between 100 per cent and the percentage of nonvolatile matter determined in Sections 22 and 24 shall be calculated and recorded as the percentage of volatile matter by weight.

(b) *Volatile Matter by Volume*.—The percentage of volatile matter by volume shall be calculated as follows:

$$x = \frac{AC}{B}$$

where:

$x$  = percentage of volatile matter by volume,

$A$  = density of the original varnish, as determined in Section 3,

$B$  = density of the volatile matter (solvent) recovered by distillation, as determined in Section 26(c), and

$C$  = percentage of volatile matter by weight, as determined in Paragraph (a).

#### Reproducibility of Results

28. With proper care and attention to detail in making this test, differences occurring between different laboratories should not exceed 2 per cent for varnishes containing solvents which have end points not over 235 C. (455 F.) and should not exceed 4 per cent when the end point of the solvent is as high as 307.2 C. (585 F.).

### DIELECTRIC STRENGTH OF VARNISH IN THE LIQUID STATE

#### Purpose

29. The purpose of the dielectric

strength test of varnish in the liquid state is to insure that the varnish is free from moisture and foreign matter such as might come from unclean shipping containers.

### Procedure

30. (a) The dielectric strength shall be determined in accordance with the method for liquids described in the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149)<sup>3</sup>, except as specified in the following Paragraphs (b) to (h) and Section 31.

(b) *Conditioning*.—The temperature of the varnish when tested shall be approximately 25 C. (77 F.)

(c) The sample of the varnish shall be shaken so that it will be thoroughly mixed before filling the test cup; the shaking shall be done gently to avoid the formation of bubbles.

(d) The cup shall be filled with varnish to a height of not less than  $\frac{3}{4}$  in. above the top of the electrodes.

(e) The varnish shall be allowed to remain in the test cup until all air bubbles have disappeared.

(f) Voltage shall be applied and increased uniformly at the rate of approximately 3 kv. (rms. value) per sec. until electrical breakdown occurs as indicated by a continuous discharge across

the gap. (Occasional momentary discharges which do not result in a permanent arc may occur; these should be disregarded.)

(g) After the breakdown and before removing the varnish from the test cup, the electrode surfaces shall be rubbed thoroughly with dry calendered tissue paper wrapped around a knife blade to provide stiffness. The test cup shall then be emptied and immediately refilled with the next filling of varnish to be tested.

(h) Five fillings shall be tested in conformity with the above procedure, and the last filling of the cup shall drain the sample container.

### Variations in Results

31. Differences as great as 15 per cent may occur between different laboratories in the results of this test. This is considered satisfactory in view of the purpose of the test.

### Report

32. The report shall include the following:

(1) Kilovolts (rms. value) at each electrical breakdown,

(2) Average value of the five breakdowns in kilovolts, and,

(3) Mean deviation from the average value expressed as a percentage of the average breakdown value.

(See Appendix, p. 512)



## APPENDIX

## DESCRIPTION AND USE OF REMODELED MACMICHAEL VISCOSIMETER FOR DETERMINING VISCOSITY OF INSULATING VARNISHES

A1. *Apparatus*.—The viscosity of varnishes may be determined by means of the remodeled MacMichael Viscosimeter shown in Figs. 1, 2 and 3. This instrument gives the viscosity values in terms of MacMichael degrees which are converted into absolute units of viscosity (centipoise) by means of the calibration curves of the various suspending wires.

(a) *Apparatus Unassembled*.—In Fig. 1 the apparatus, consisting of the following parts, is shown unassembled:

*A* is the indicator by means of which the zero is adjusted and readings on scale *D* are taken.

*B* is the insulated wire suspension, the torsion of which measures the viscosity. The wire is fastened only at the bottom of its insulating sleeve *E'*. This is suspended by hanging it on points *P'*. (Ten wires of different size to cover various ranges of viscosities are supplied.)

*C* is the clamp screw which clamps the hollow spindle *E* to the sleeve *E'* which holds the torsion wire *B*.

*D* attached to hollow spindle *E* is the dial on which are calibrated the degrees MacMichael (300 deg. to the circumference). By means of this dial and indicator *A* the degrees deflection are determined as indicated in the method described below.

*E* is the hollow vertical spindle inside of which is the torsion wire sleeve *E'* which contains torsion wire *B*.

*F* is an oil-filled dash pot which acts as a brake to deaden oscillations and bring the suspension to rest quickly when making a determination. The dash pot swings between arm guides *F'* when the machine is assembled.

*G* is the brass disk which is attached to the bottom of vertical spindle *E*. Torsion wire *B* and *E'* is inserted into *E* and clamped with screw *C* and the disk *G* is then suspended in the liquid whose viscosity is being measured. Rotation of the sample cup *H* rotates the liquid and the rotating liquid exerts a certain force on the disk *G* partially rotating it and thus a torsion is produced in the wire suspension *B*, the deflection of which (or degree of torsion) is read from the scale on deflecting disk *D*.

*H* is the container which holds the sample. A locking device fastens it securely to *I* or *J*.

*I* is the water bath used for securing a uniform temperature in the liquid whose viscosity is being determined, when these viscosity meas-

urements are desired at temperatures approximating that of the room (used on varnishes and oils).

*J* is the heating unit which is used in place of *I* when viscosity measurements are made at high temperatures (used on compounds).

*K* is the contact which carries the electric current to the heating unit cup *J*. It can be swung back when using cups *H* and *I* and when therefore electric heating is not desired.

*L* is the counter which indicates the revolutions per minute of the turntable *M*. A trip arrangement allows this counter to be lowered or raised, thus throwing the counter in or out of operation at will.

*M* is the rotating turntable in which *I* or *J* are set. A locking device (not shown) locks *I* or *J* securely to the turntable *M* so that when the turntable rotates the water bath or heating unit with the sample container also rotate.

*N* is the enclosing case under which is the motor and governor shown in Fig. 3. These operate turntable *M*.

*O* is a rheostat which controls the current going to the heating unit *J*.

*O'* is a rheostat controlling current for motor (shown only on remodeled instruments).

*P* is the knife switch (snap switch on new type machine) controlling the current for the heating unit *J*.

*R* is the push button which controls the electric current operating the motor shown in Fig. 3.

*S* is a leveling screw to level the instrument. Another such screw (not shown) is directly opposite to this one.

*T* is the governor screw that adjusts the speed of the motor by adjusting the governor shown in Fig. 3. In the remodeled apparatus the speed of rotation of turntable *M* can be decreased by moving the large gear in gear box away from the motor until pin of motor shaft is disengaged from slot of gear sleeve. The instrument is sent out with gear in low-speed position; to obtain high speeds shift gear toward motor until pin is engaged.

*U* is the measuring device used to measure the depth of the liquid in container *H*.

*p* are the projecting points on which the assembled spindle, namely, insulated torsion wire suspension *B* and *E'*, spindle and deflection dial *D*, and immersed disk *G* are suspended.

(b) *Apparatus Assembled for Test*.—The apparatus is shown assembled in Fig. 2 for viscos-

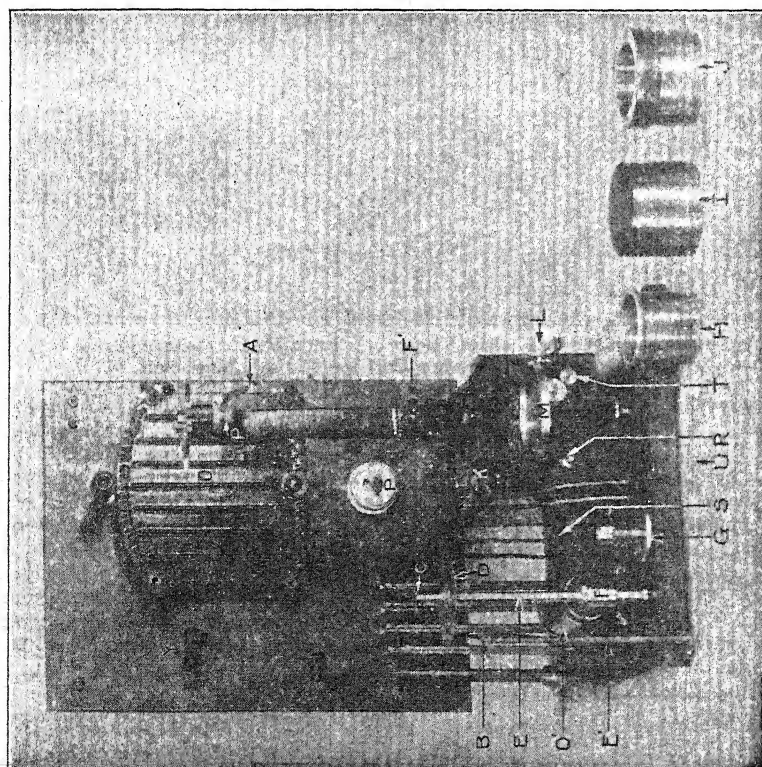


FIG. 1.—Improved MacMichael Viscosimeter Apparatus, Unassembled.

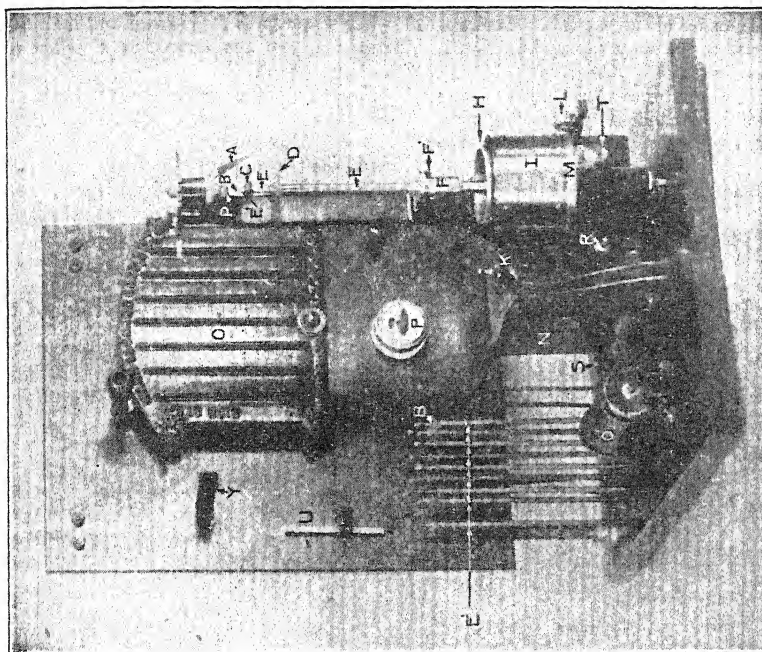


FIG. 2.—Improved MacMichael Viscosimeter Apparatus Assembled for Testing Varnish or Oil.

ity determinations on oil or varnish. Explanatory letters refer to the same parts as in Fig. 1.

(c) *Internal Structure of Apparatus.*—In Fig. 3 are shown in the internal structure and cup and heating unit assembled for testing compounds. The explanatory letters refer to the following:

*V* is the motor which operates the turntable *M*.

*W* is the governor controlling the speed of the turntable *M*.

*X* are the leads to connect instrument to 110-v. a-c. or d-c. lighting circuit.

Use water bath *I* to secure and maintain the temperature of the sample in *H* when testing varnishes or oil.

Insert container *H* into water bath *I*. See that they are properly locked together, then lock bath *I* on turntable *M*.

Choose the proper torsion wire suspension for the liquid to be tested. Experience is the best guide to determine what suspension to use. Screw the disk *G* to the spindle *E*, and insert into *E* the proper insulated torsion wire suspension *E'*. Suspend these assembled parts from points *p* so the disk is suspended in the

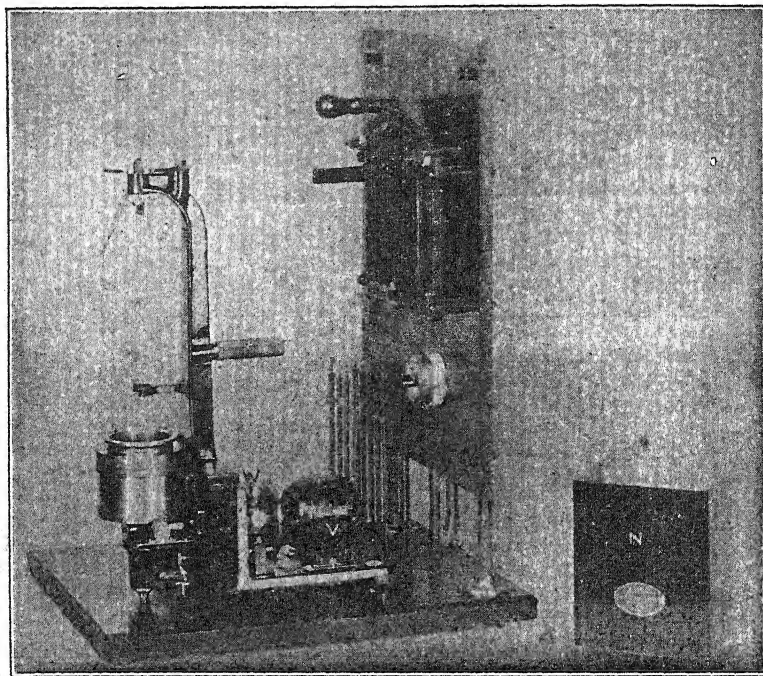


FIG. 3. Improved MacMichael Visosimeter Apparatus Showing Internal Structure and Cup and Heating Unit for Testing Compounds.

*Y* is the clamp holder for holding the spindle *E* with its dial *D*, when the machine is not in use (remodeled machines).

The heating unit *J* and the sample container *H* are here shown assembled as used in the viscosity determinations on compounds.

A2. *Directions for Making Viscosity Measurements on Varnish.*—Cups and disks must be perfectly clean. Otherwise reliable results cannot be obtained.

Dash pot *F* on the stem of the spindle should be two-thirds full of a medium grade of good machine oil (Etna Engine or Appollo) in order to dampen vibrations.

container *H*. Be sure always to suspend the wire on the points *p* with the flat side of the triangular button against the flat side of the supporting arm. The rounded surface of the triangle must face out towards the operator. Tightly fit the cover of the dash pot *F* on the arm guides *F'*.

See that the instrument is perfectly level. Use leveling screws *S* to adjust the instrument. The instrument is level when the suspended sleeve *E* swings exactly in the center of the hole of the cover on the dash pot.

Adjust the speed of the turntable to 60 rpm. This should be checked each time de-

terminations are made. Use the governor screw *T* to adjust the speed of the motor. The gear in the gear box, referred to under *T* in the description of Fig. 1, should be in high speed position, that is, towards the motor. Use a stop watch to adjust the speed. After the speed is adjusted, lower the counter *L* so it will not operate during the following procedure.

NOTE.—The deflection is directly proportional to the speed of rotation. The 60 rpm. must therefore be maintained when the varnish, oil or compound is in the cup. It is, therefore, necessary to check first the revolutions per minute without the disk suspended in the sample, and again, when running a determination with the disk suspended in the sample. The motor must give the same number of revolutions per minute in each case and this value must be the same as was used for calibrating the suspension.

When the instrument is level, adjust the indicator *A* to the zero mark on the dial *D*, when the spindle is suspended in the air, by moving the indicator as necessary. When the indicator has been set it must not be moved again until the test has been completed.

Fill the container *H* with the sample to be tested to a depth of 3.5 cm. A special device *U* is provided to measure this depth. Lay this device across the cup so the pointed end projects into the cup. When the surface of the liquid just touches the point, the depth of the liquid in the cup is 3.5 cm.

Raise or lower the temperature of the oil or varnishes to 21 C. by adding hot or cold water to the water bath, always stirring the sample well until a uniform temperature is secured. This is imperative. The sample may be stirred by rotating the sample cup frequently. It may also be stirred by lifting the spindle from the points *p* and stirring with the disk *G*, holding the spindle by the barrel *E* and stirring by giving the disk an up-and-down movement in the sample cup. In the case of compounds, it is necessary to stir in this manner. In testing varnishes and oils, usually sufficient stirring can be obtained by rotating the cup while holding the barrel *E* to keep the disk stationary. If the spindle is removed from the points *p*, it must always be suspended again with the same flat side of the supporting triangular button against the supporting arm so that the zero position is not changed.

If when resuspending the spindle from points *p* it is noted that the zero point has moved, do not reset the indicator *A* as the change probably is due to some torsional effect produced by the sample. This may be especially noticed when

testing compounds. Rotate the cup and take the reading without resetting the indicator.

When reading the thermometer, have the bulb in the sample midway between the disk and the container wall and not resting on the bottom. Test the temperature at various points in the container to determine whether a uniform temperature has been secured.

When a uniform temperature of 21 C. is secured, start the cup rotating by closing the switch *R*. Keep a hand on the switch and watch the scale on the disk *D*. If there is any danger that the deflection will go beyond the maximum limit for the suspension being used, open the switch immediately. Insert the next strongest wire suspension into the spindle and repeat as above indicated. When changing suspensions it is necessary to again set for zero. This may be accomplished without removing the sample by simply removing the water bath *I*, with its

TABLE II.—MAXIMUM AND MINIMUM LIMITS FOR SUSPENSIONS.

Suspension Number (B. & S. Gage No.)	Minimum Deflection, deg. MacMichael	Approximate Equivalent Centipoise Reading	Maximum Deflection, deg. MacMichael	Approximate Equivalent Centipoise Reading
30 (Weakest)....	30	18	125	89
28.....	20	35	100	200
26.....	7.5	30	35	175
24.....	5	70	32.5	400
22.....	10	200	45	975
21.....	5	225	40	1800
20.....	10	750	50	4200
18.....	4	700	24	4000
16.....	2	750	11	4000
14 (Strongest)...	1	1375	5	5500

sample container *H*, and then suspending the spindle with its new suspension on *p*, and leveling and adjusting as described above. It is necessary, of course, to thoroughly clean the disk *G* when doing this, for if any varnish film should dry on the disk, the uneven or rough surface thus produced on the disk would cause an erroneous reading. A suspension wire should never be allowed to deflect beyond its maximum limit, for beyond this point a permanent set would be produced in the wire and a new calibration of the wire would have to be made or probably a new wire would have to be used. Table II gives a maximum and minimum limit for suspensions now in use. If the deflection is less than this minimum limit, the next weakest suspension must be inserted and the test repeated. The reason for this is that at low deflections a change in slope appears on the calibration curve of the MacMichael degrees *versus* centipoises. The percentage error is magnified here, and hence, a reading that

comes up higher in the calibration curve should be used. The maximum limit given may not be the limit to which it is permissible to deflect the wire in order to prevent its acquiring a permanent set. It, however, happens to be the limit of the calibration curve available for that suspension when the wire was calibrated.

Record the deflection in degrees MacMichael and read the viscosity in centipoises from the calibration curve of the suspension used.

NOTE 1.—Before any sets of suspensions are used, they must be calibrated as described in Paragraph A3.

NOTE 2.—The following table gives the diameter of the wires (new American music wire gage) used in making the suspensions and the maximum limit of torsion in degrees MacMichael to which they may be subjected before they acquire a permanent set:

Suspension Number (B. & S. Gage No.)	Diameter of Wire, in.	Torsional Limit, deg. MacMichael
30 (Weakest).....	0.0100	125
28.....	0.0126	118
26.....	0.0159	110
24.....	0.0201	102
22.....	0.0254	92
21.....	0.0285	81
20.....	0.0320	73
18.....	0.0403	55
16.....	0.0508	35
14 (Strongest).....	0.0641	5

NOTE 3.—Not all of the suspensions noted in the table in Note 2 are required in testing varnish. Suspensions 22, 21, and 20 will be sufficient for practically all varnishes. The heavier wires 18, 16, and 14 are used for very viscous materials.

A3. *Calibration of Suspensions.*—For the calibration of the torsion-wire suspensions used in this apparatus to measure viscosity, samples of oils verified as to absolute viscosity at 25 C. are available from the National Bureau of Standards. A sufficient number of oils to cover the range of viscosity desired should be procured. New samples should be obtained at least once a year as it has been found that the oils undergo some change on standing over long periods. The calibration of the wires is made by testing the certified samples of oils at  $25 \pm 0.2$  C., following the directions given in Paragraph A2.

The degrees MacMichael obtained for each suspension on each sample are plotted against corresponding National Bureau of Standards absolute viscosity figures (centipoise) for those samples and a line is drawn through these points. The line should be straight except possibly at low deflections where the error is correspondingly high. Once every two months the suspension should be recalibrated.



# Tentative Methods of

## TESTING VARNISHED CLOTHS AND VARNISHED CLOTH TAPES USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 295 - 46 T

ISSUED, 1928; REVISED, 1929, 1932, 1933, 1934, 1936, 1937, 1938, 1943, 1944, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover procedures for the testing of varnished cloths and varnished cloth tapes (Note) to be used as electrical insulation and are directly applicable to both the "straight-cut" and "bias-cut" materials, unless otherwise stated in the method.

NOTE.—Methods of testing varnished glass fabrics and tapes are given in the Tentative Methods of Testing Varnished Glass Fabrics and Varnished Glass Fabric Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 902).<sup>3</sup>

### SAMPLING

#### Selecting Rolls

2. (a) Shipments of varnished cloth and varnished cloth tapes shall be sampled as specified in Paragraphs (b) and (c). The rolls or pads shall be selected in such a manner as to be representative of the shipment.

(b) *Cloth*.—One roll shall be selected from each ten rolls or fraction thereof in a shipment of full width cloth.

(c) *Tape*.—Rolls of tape shall be se-

lected from each shipment in accordance with the following schedule:

Number of Rolls in Shipment	Minimum Number of Sample Rolls
Over 10 000.....	1 per thousand
5 001 to 10 000.....	10
2 001 to 5 000.....	5
501 to 2 000.....	2
200 to 500.....	1
Less than 200.....	optional

(d) Samples of oil-immersed tape shall be taken from not less than two cans.

#### Selecting Samples

3. (a) Except for tape shipped packed in oil, not less than two turns of cloth or six turns of tape from each roll or pad selected for sampling shall be torn off and discarded before the samples are selected. The number of samples required for purpose of tests shall then be cut across the entire width of each roll or pad. The length of the samples shall be not less than 36 in. (91 cm.) for straight-cut material, from seam to seam for sewed bias-cut material, and from jointed-selvage to jointed-selvage for seamless bias-cut material.

NOTE 1.—Specimens of bias-cut materials cut from the above samples shall exclude seams and jointed-selvages from the test areas.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A S.T.M. Designations at front of book.

NOTE 2.—If it is desired to test the seams or jointed-selvages for breaking strength, additional specimens will have to be cut so that the seams or jointed-selvages are in the center of the specimens.

(b) From shipments such as sheets of cloth or strips of tape, the samples shall be representative of the shipment and shall be taken in accordance with Paragraph (a).

#### CONDITIONING

##### Conditioning

4. (a) If it is desired to test the material in the condition as received by the purchaser, the test samples shall not be specially conditioned, but the packages containing the rolls of cloth or tape from which the samples are to be taken shall be allowed to reach approximately test room temperature before the packages are opened and the samples cut. Specimens to be tested shall be removed from the roll as required and tested immediately, unless otherwise specified.

(b) Where it is desired to test in a controlled atmosphere, the test specimens shall be conditioned for 48 hr. in air maintained at a temperature between 68 and 86 F. (20 and 30 C.) and at a relative humidity of  $50 \pm 2$  per cent.

(c) If a conditioning cabinet or chamber is used, test specimens shall be subjected to test immediately upon withdrawal from the cabinet or chamber, unless otherwise specified.

#### THICKNESS

##### Test Specimens

5. Test specimens shall consist of samples selected in accordance with Section 3.

##### Procedure

6. The thickness (Note) shall be measured in accordance with Method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation

(A.S.T.M. Designation: D 374),<sup>3</sup> modified as follows:

(1) In making thickness measurements, only one layer of material shall be used.

(2) In the case of cloths, ten measurements equally spaced across the width of the specimen shall be taken and the thickness of the cloth shall be the average of the ten measurements.

(3) In the case of tapes, unless otherwise specified, ten measurements equally spaced along the length of each specimen shall be taken and the thickness of the tape shall be the average of the ten measurements.

NOTE.—The average thickness, as obtained for the dielectric strength test (Section 14), may be conveniently used instead of the separate test described in Section 6.

##### Report

7. The report shall include the average, maximum, and minimum thicknesses in inches.

#### BREAKING STRENGTH

##### Apparatus

8. (a) A testing machine<sup>4</sup> of the dead-weight pendulum type shall be used. The machine preferably shall be power driven.

(b) The capacity of the machine shall not exceed 250 lb. (113 kg.).

(c) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing specimens breaking at 50 lb. (22.7 kg.) or over, and to 0.5 lb. or 0.5 kg. or less for testing specimens breaking under 50 lb. (22.7 kg.).

##### Test Specimens

9. Specimens cut from the full width

<sup>4</sup> This apparatus is the same as that used for the testing of paper and is described in Section 10 of the Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (A.S.T.M. Designation: D 202), see p. 685.

cloth or from tapes over 1 in. (2.54 cm.) in width shall be 1 in. (2.54 cm.) in width and not less than 14 in. (35.5 cm.) in length. For tape having a nominal width of 1 in. (2.54 cm.) or under, the specimens shall be of the original width and shall be not less than 14 in. (35.5 cm.) in length.

NOTE.—If it is desired to test the seams or jointed-selvages of bias-cut materials for breaking strength, specimens obtained as described in Note 2, Section 3 (a) shall be clamped in the testing machine so that the seams or jointed-selvages are midway between the two jaws. When testing, all readings obtained when the specimen breaks away from the seam or jointed-selvage shall be rejected.

#### Number of Specimens

10. (a) In the case of straight-cut cloths, ten specimens shall be cut in the direction of the warp threads and ten in the direction of the filling threads from samples selected in accordance with Section 3.

(b) In the case of bias-cut cloths, ten specimens shall be cut in the direction of the length from samples selected in accordance with Section 3.

(c) In the case of tapes, one specimen shall be cut from each roll selected in accordance with Section 2 (c).

#### Procedure

11. (a) The ratio of the clearance distance between jaws to the width of the specimen shall be not less than 5 to 1 nor more than 10 to 1.

(b) The rate of travel of the movable jaw shall be constant and preferably 12 in. (30.5 cm.) per min., but it may be within the limits of 11 and 13 in. (28 and 33 cm.) per min., provided it is constant.

(c) All readings obtained when the specimen breaks at or in the jaws shall be rejected.

#### Report

12. (a) The breaking strength of a

roll shall be the average of the breaking loads of all the specimens tested from the roll. The report shall include the average, maximum, and minimum breaking loads in pounds or kilograms together with the width of the specimens and the nominal thickness.

(b) In the case of straight-cut cloths, the breaking strength of the warp threads and the filling threads shall be reported separately.

#### DIELECTRIC STRENGTH

##### *Short-Time and Step-by-Step Tests*

#### Purpose

13. The purpose of this test is to determine the dielectric strength of varnished cloths and varnished cloth tapes when subjected to a short-time or step-by-step test.

#### Dielectric Strength

14. (a) The dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials,<sup>3</sup> except as specified in the following Sections 15 to 20.

(b) Dielectric strength tests may be applied to specimens of the material as-received, and on specimens from the elongation, resistance to oil, and humidity tests.

#### Apparatus

15. For the testing of narrow specimens, such as tapes, it is necessary to use a special form of testing device, whereby the test specimen shall be clamped under pressure in order to prevent flashover around the edges of the material. Two forms of testers are described in the Appendix.

#### Test Specimens

16. (a) In the case of cloths, a speci-



men at least 4 in. (10.16 cm.) in width shall be cut diagonally from corner to corner from a sample selected in accordance with Section 3.

(b) In the case of tapes, the specimens shall be samples selected in accordance with Section 3.

#### Surrounding Medium

17. Tests shall be made in air unless the material is to be used under oil, in which case the material may be tested under oil.

#### Conditioning

18. Specimens shall be conditioned in accordance with Section 4.

#### Procedure

19. (a) Tests shall be made by either the short-time test or the step-by-step test, or by both methods.

(b) In the short-time test, voltage shall be increased at the rate of 0.5 kv. per sec.

(c) In tests made by the step-by-step test, each step shall be of 20-sec. duration. Voltage shall be increased by the following increments:

Nominal Thickness of Tape	
8 mils or less.....	250 v.
Over 8 mils.....	500 v.

The starting voltage shall be adjusted to the nearest even 250 or 500 v., depending on the increment of increase. The starting voltage should preferably be based upon the breakdown voltage in the short-time test, as provided in A.S.T.M. Methods D 149. Where this may not be feasible or convenient, a starting voltage may be selected as the result of test experience, of such a value that at least three testing steps are obtained prior to breakdown.

#### Report

20. The report shall include the following:

(1) The test method used for determining the dielectric strength,

(2) The average dielectric strength in volts per mil and any other values of dielectric strength as required by the materials specifications, and

(3) The temperature at which the tests were made.

#### ENDURANCE DIELECTRIC STRENGTH

##### Purpose

21. The purpose of this test is to obtain some idea of the relative dielectric strength of various materials when subjected to electrical stress for relatively long periods at high temperature.

##### Test Specimens and Apparatus

22. (a) The specimens shall be made by wrapping the material in tape form with one-quarter lap, one-half lap, or butt-jointed, or in sheet form, on brass tubes. For sheets and for tape up to 1½ in. in width, the tubes shall be about 36 in. in length and 1 in. in outside diameter. For tapes wider than 1½ in., the tubes shall be 2 in. in outside diameter. The sample shall be built up to the desired thickness, simulating practical conditions, winding all layers of tape in the same direction.

(b) A smooth layer of metal foil shall then be wrapped over the insulation for a distance of 24 in., leaving 6 in. of insulation uncovered at each end of the tube. The metal foil shall be bound in place with a wrapping of adhesive tape which shall extend the full length of the tube in order to protect the insulation at the end from corona discharge.

(c) A thermocouple shall be applied to the metal foil near the center of the tube and held in place by the adhesive tape.

##### Procedure

23. (a) The specimen shall be mounted

in an oven and a temperature of 100 C. (212 F.) maintained during the test. Provision shall be made for so mounting the specimen that the tube may be connected to the high voltage side of the circuit, with the metal foil sheath and thermocouple connected to ground.

(b) A voltage equal to 10 per cent of the breakdown voltage (to the nearest kilovolt) obtained in the short-time test shall be applied and maintained for 30 min. The voltage shall then be increased by steps of 20 per cent of the initial value until puncture occurs, the voltage being held at each step for 30 min.

(c) The temperature of the sample as indicated by the thermocouple shall be observed at intervals during the test and recorded at the end of each 30-min. period. It will be found that the temperature rises gradually until just before breakdown when the temperature increases rapidly. During this latter period, temperature readings shall be recorded at frequent intervals.

### Report

24. (a) The results shall be plotted using time, expressed in hours, as abscissas, and temperature, in degrees Centigrade, as ordinates. In addition to time as abscissas, the values of applied kilovolts shall be indicated, since the value of kilovolts increases uniformly with time. The significant values to be taken from the curve and reported areas follows:

- (1) Duration of test,
- (2) Breakdown voltage,
- (3) Temperature of specimen at breakdown, and
- (4) Rate of temperature rise during test.

(b) The report shall also include a description of the specimen and its preparation, the thickness of the insula-

tion, and the number of layers of insulation.

### ELONGATION

*(In the Case of Bias-Cut Material)*

#### Purpose

25. The purpose of this test is to determine the tendency of bias-cut varnished cloths and tapes to stretch under mechanical tension. This test is not applicable to tapes of less than 0.5 in. (1.27 cm.) in nominal width.

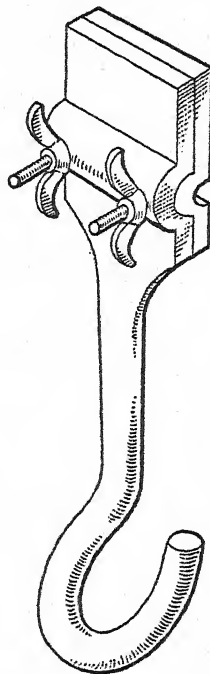


FIG. 1.—Clamp for Elongation Test.

#### Apparatus

26. The apparatus shall consist of a pair of clamps for gripping the ends of the specimen. The clamps shall be not less than 2.0 in. (5.08 cm.) in width, and one shall be provided with means for attaching to a fixed support and the other with means for affixing suspended weights. A suggested form of clamp is shown in Fig. 1.

### Test Specimens

27. (a) In the case of bias-cut cloths, specimens shall be cut in the direction of the length from the samples selected in accordance with Section 3. They shall not exceed 1.5 in. (3.8 cm.) in width and shall be at least 36 in. (91 cm.) in length.

(b) In the case of bias-cut tapes, specimens shall be samples selected in accordance with Section 3 and they shall not exceed 1.5 in. (3.8 cm.) in width. Tapes of greater width than this shall be cut 1.5 in. (3.8 cm.) or less before testing.

### Procedure

28. (a) A gage length of 50 cm. shall be marked on the test specimen (Note) and the specimen shall be fastened between two suitable clamps so that the gage length is centrally located between the clamps. The clearance distance between each gage line and the adjacent clamp shall be not less than 10 cm.

NOTE.—Strips of Scotch cellulose tape make a satisfactory method for marking the gage length on the elongation test specimens.

(b) The test specimen shall be weighted with a specified load  $M$  (including the weight of the clamp) and at the end of 35 min. the distance  $L$  between gage lines shall be measured and the load then removed.

### Calculation

29. The percentage elongation shall be calculated as follows:

$$\text{Elongation, per cent} = \frac{L - 50}{50} \times 100$$

where:

$L$  = distance in centimeters between gage lines at end of the test.

### Report

30. The report shall include the following:

(1) the load in pounds per inch width at tape,

(2) the percentage elongation, and

(3) the temperature of the room during the elongation test.

### DIELECTRIC STRENGTH UNDER ELONGATION

#### Purpose

31. The purpose of this test is to determine the effect of elongation on the dielectric breakdown of bias-cut varnished cloths and tapes. This test is not applicable to tapes of less than 0.75 in. (1.905 cm.) in nominal width.

#### Apparatus

32. (a) The apparatus shall consist of a pair of clamps for gripping the ends of the specimen and some suitable means of securing the clamps so that they can be pulled away from each other at an approximately uniform rate. For this purpose, a hand or motor-operated pulling outfit or a tensile-testing machine of suitable range may be used.

(b) A special form of dielectric tester, as described in Section 15, shall be used for making breakdown tests. The tester shall be arranged to receive the test specimens while the latter are held in the stretched condition. Where a special pulling outfit is used, this may be so set up that the tape is pulled through the separated electrode blocks of the tester. When a tension-testing machine is used, it may be more convenient to transfer the test specimen, while under tension, to a special frame which may be readily inserted into the tester.

#### Test Specimens

33. Test specimens shall be prepared as described in Section 27.

#### Procedure

34. (a) A gage length of 50 cm. (19.68 in.) shall be marked on the test specimen

and the specimen shall be fastened between the clamps so that the gage length is centrally located between the clamps. The clearance distance between each gage line and the adjacent clamp shall be not less than 10 cm. (3.9 in.).

(b) The clamps shall then be moved apart at a rate of approximately 5.08 cm. (2 in.) per minute until the tape has been elongated 6 per cent. After this elongation has been reached, it shall be maintained for a minimum of 5 min. after which five breakdowns shall be made by the short-time test described in A.S.T.M. Methods D 149. The five tests shall be distributed uniformly within the gage length, and shall not consume more than 10 min. of elapsed time. The dielectric strength shall be calculated on the basis of the average thickness of the specimen determined immediately after the dielectric breakdown test and while the specimen is elongated.

NOTE.—In the use of tape it is probable that it may be elongated more than 6 per cent. In order to obtain dielectric strength data at a higher percentage elongation, it is recommended that tests be made at 12 per cent elongation in addition to the specified 6 per cent elongation.

#### Report

35. The report shall include the following:

(1) The average dielectric strength in volts per mil on the elongated material.

(2) The temperature at which the tests were made.

#### POWER FACTOR AND DIELECTRIC CONSTANT

##### Purpose

36. The purpose of this test is to determine the power factor and dielectric constant of varnished cloths and varnished cloth tapes at frequencies not exceeding 70 cycles per second.

#### Definitions and Theory of Test

37. The definitions of dielectric power factor, dielectric constant, and related terms, and the theory of test shall be as given in the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup>

#### Electrodes

38. Flat, rigid, guarded electrodes, not over 10 sq. in. in area, of such size as to give the bridge sufficient sensitivity to detect readily a change in power factor of 0.0005, shall be used. The specimen shall extend beyond the guard electrode for a distance of at least four times the thickness of the specimen. The pressure shall be not less than 10 nor more than 20 psi.

#### Test Specimens

39. The test specimen may be tested in one thickness. Petrolatum may be used to avoid ionization. The petrolatum shall have a power factor at 80 C. of not exceeding 0.02 at 60 cycles, or a resistivity not less than  $10^{12}$  ohms per centimeter cube at 80 C.

#### Conditioning

40. The test specimens shall be conditioned by one of the following two methods:

(a) When the greatest reproducibility of results is desired, the test specimens in one thickness shall be heated to  $105 \pm 3$  C. for 1 hr. without vacuum and then for 2 hr. at  $105 \pm 3$  C. in a vacuum having an absolute pressure not exceeding 1 mm. of mercury.

(b) Where the vacuum treatment is not feasible or where tests in the as-received condition are desired, or where an approximation to some conditions of use is desired, the test specimens shall

be conditioned in accordance with Section 4.

(c) In the case of dispute the vacuum treatment shall be considered the referee method.

#### Procedure

41. (a) Tests shall be made at room temperature of 25 to 30 C., and at  $80 \pm 1$  C. The conditioned specimens shall be tested within 5 min. after removal from the vacuum container. The voltage gradient shall be 50 v. per mil. The method of measurement shall conform to that described in A.S.T.M. Methods D 150.

(b) Three specimens shall be tested and the values obtained averaged.

#### Report

42. The report shall include the following:

(1) *Test Conditions.*—The frequency in cycles per second, the temperature in degrees Centigrade, relative humidity in per cent, the conditioning of the specimen, size of electrodes, pressure on electrodes, and voltage gradient in the dielectric while under test.

(2) *Test Specimen.*—Capacitance of specimen in micro-microfarads, effective area of specimen electrodes, and average thickness of specimen between electrodes.

(3) Power factor, dielectric constant, and loss factor of each specimen, and the average.

(4) Method of measurement.

### INSULATION RESISTANCE

#### Procedure

43. The insulation resistance of varnished cloths and varnished cloth tapes shall be determined in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation:

D 257).<sup>3</sup> The test specimen shall be prepared in accordance with Fig. 1 of A.S.T.M. Methods D 257.

### RESISTANCE TO OIL

#### Purpose

44. The purpose of this test is to determine the effect of oil on the varnish film and dielectric strength of the cloths or tapes.

#### Test Specimens

45. One specimen 12 in. (30.5 cm.) in length and not exceeding 1.5 in. (3.8 cm.) in width shall be cut from each of the rolls selected in accordance with Section 2.

#### Procedure

46. The specimens shall be immersed for 15 min. in a specified oil maintained at a temperature of from 97 to 103 C. At the end of this time, the specimens shall be removed from the oil, allowed to cool for at least 30 min. to room temperature, and any excess oil removed by placing the specimens between blotters without any sliding.

#### Examination

47. The varnish film shall be examined for disintegration in the oil and flaking either in the oil or on the blotter. Disintegration in the oil may be detected by examination of the used oil for turbidity.

NOTE.—The oil may be considered turbid if a sample of used oil filtered through filter paper is distinctly less transparent than an unfiltered sample of the unused oil when the two samples, in identical containers, are held in front of a diffused light. Flaking along the cut edges of tapes shall not be considered as disintegration of the varnish film.

#### Dielectric Strength

48. Specimens which have been allowed to cool at least 30 min. may be

tested for dielectric strength any time within a period of 4 hr. after removal from the oil. Five puncturing voltages shall be taken on the specimens in accordance with Section 14, using only the short-time test described in A.S.T.M. Methods D 149. The dielectric strength shall be calculated on the basis of the average thickness of the specimen determined immediately before the dielectric breakdown test.

#### Report

49. The report shall include the following:

(1) The type of oil used (preferably including the flash point determined in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92) of the American Society for Testing Materials),<sup>5</sup>

(2) The temperature of the oil,

(3) Results of the physical examination of the film (Section 47),

(4) The percentage increase in average thickness due to oil tests, and

(5) The average dielectric strength in volts per mil after the oil tests.

#### WEIGHT

##### Procedure

50. Specimens of sufficient size to weigh not less than 5 g. (0.18 oz.) shall be accu-

rately weighed on an analytical balance and the weight shall be reported on some commercial unit basis such as the weight per square yard, the weight per gross yard per 1 in. in width, or the weight per roll.

#### THREADS PER INCH

##### Procedure

51. The count or threads per inch shall be determined separately on the warp and filling by counting in a space of not less than 1 in. in at least five different places in the sample strip (or strips). The average of the five determinations shall be the count.

##### Report

52. The results of the warp count and the filling count shall be reported separately as threads per inch.

NOTE 1.—The warp threads in straight-cut materials are the threads that are parallel with the length dimension, while in bias-cut materials the warp threads are parallel with the seams and jointed-selvages.

NOTE 2.—Before counting black varnished materials it will be necessary to remove the varnish film with a knife blade or other suitable instrument. Liquid varnish removers are unsuitable for this purpose as they cause a swelling of the fibers and a shrinkage of cloth with a consequent increase in threads per inch; therefore, the films must be removed mechanically.

<sup>5</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

## APPENDIX

## APPARATUS FOR MAKING DIELECTRIC STRENGTH TESTS ON INSULATING TAPE

A1. *Single Shot Tester*.—Figure 2 illustrates a single-shot tester for making dielectric strength tests on insulating tape. In this device the tape is held under pressure between rubber washers while the voltage is applied, the pressure being supplied by compressed air operating on a piston *b* in a cylinder *a*. The piston is connected to the upper electrode *c*. The lower electrode *c'* is mounted on the insulated base. The electrodes terminate in  $\frac{1}{2}$ -in. brass rods

This lower assembly is also free to rock very slightly on a single ball support. This insures perfect contact and equalization of pressure when the electrode assemblies come together.

The upper electrode assembly is normally held clear of the lower assembly by two small springs so that tape may be readily inserted or withdrawn or moved to a new position of test. The piston *b* is smooth, without rings, and permits just enough leakage of air so that the pres-

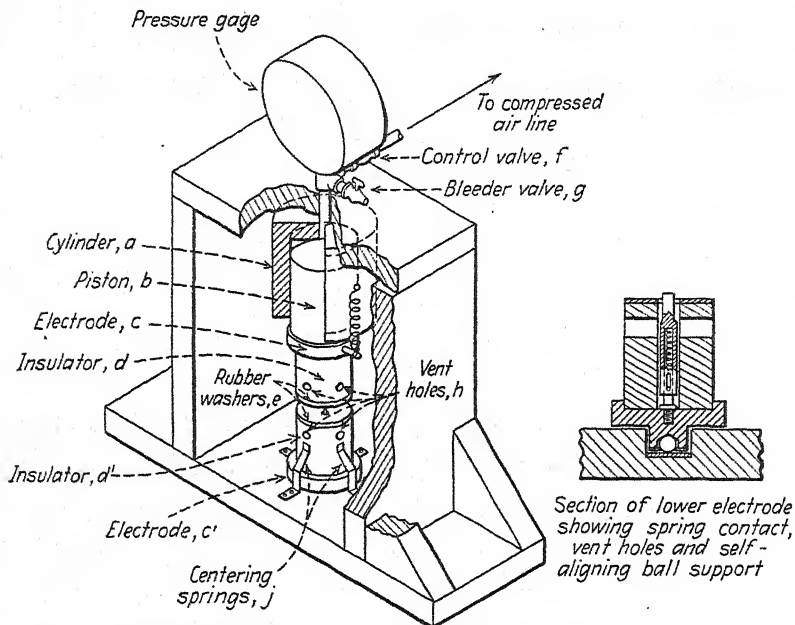


FIG. 2.—Single-Shot Tester for Dielectric Strength Test on Insulating Tape.

having flat ends with edges rounded to a radius of  $\frac{1}{32}$  in. These electrode rods move against light springs. This insures a positive contact between electrode and tape. The electrode rods are surrounded by insulating blocks *d* and *d'* having vent holes *h* for the dissipation of the gases generated by breakdown. These insulating blocks are faced with soft rubber washers *e* which can be detached and replaced as necessary.

The lower electrode assembly is lined up with the upper electrode assembly by centering springs *j*, the position of which is adjustable.

sure may be quickly adjusted and readily held at any desired value below the maximum available.

Where compressed air is not available, pressure may be exerted by a hand-operated lever attached to the upper, or movable, electrode assembly.

A2. *Multiple Electrode Tester*.—Another form of tester, known as the multiple-electrode type, clamps a long piece of tape under pressure between two insulating blocks and has ten sets of electrodes spaced at intervals along the test specimen.



# *Tentative Methods of*

## TESTING MOLDED MATERIALS USED FOR ELECTRICAL INSULATION<sup>1</sup>



**A.S.T.M. Designation: D 48 - 46 T**

ISSUED, 1941; REVISED, 1942, 1943, 1945, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover a compendium of tests which apply to solid electrical insulating materials formed by a molding operation employing pressure with or without heat, and of instructions and recommendations supplementary thereto which apply to these particular materials. Dry-process porcelain is not included. Primarily these methods give instructions for preparing the materials for test and any choices or modifications to be made in general test methods which otherwise are to be followed exactly. The test methods included are listed in Table I. Information on the utility of these tests is given in the Appendix.

### MOLDING OF TEST SPECIMENS

#### Molds

2. Test specimens shall be molded in molds conforming to the Tentative

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

Prior to their present publication as tentative, these methods were published as tentative from 1917 to 1922, being revised in 1921. They were adopted in 1922, published as standard from 1922 to 1942, being revised in 1924, 1927, 1929, 1930, 1933, 1937, and 1939, but withdrawn as standard in 1942.

TABLE I.

Test	Preparations for Test Described in These Methods in Section No.	A.S.T.M. Designation of Test Method
Acetone Extraction (phenolic materials)	42	D 494
Arc Resistance (thermosetting materials)	8, 9	D 495
Brittle Temperature (nonrigid plastics)	31	D 746
Chemical Resistance	41	D 543
Conditioning	4-7	D 618
Deformation Under Load (except nonrigid plastics)	29	D 621
*Dielectric Constant	19, 20	D 150
*Dielectric Strength	10-15	D 149
Distortion Under Heat (except nonrigid plastics)	32, 33	D 648
Flammability (except nonrigid plastics)	36, 37	D 635
Flow Temperature (thermoplastics)	44	D 569
*Insulation Resistance	16-18	D 257
*Loss Factor	19, 20	D 150
Mobility (thermosetting materials)	45	D 731
Molds for Test Specimens	2	D 647
Powder Tests	43	D 392
*Power Factor	19, 20	D 150
Shrinkage	46	D 551
Stiffness (nonrigid plastics)	30	D 747
Strength:		
Compressive (except nonrigid plastics)	21, 22	D 695
Flexural (except nonrigid plastics)	23, 24	D 790
*Impact (except nonrigid plastics)	25, 26	D 256
*Tensile (nonrigid plastics)	27, 28	D 412
*Tensile (thermoplastic)	27, 28	D 638
*Tensile (thermosetting and cold-molded)	27, 28	D 651
Thermal Expansion, Coefficient of		
Linear (except nonrigid plastics)	34, 35	D 696
*Volume Resistance	16-18	D 257
Water Absorption	38-40	D 570

\* A discussion of the significance of these tests appears in the special publication, "A.S.T.M. Standards on Electrical Insulating Materials," October, 1945, p. 27.



Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (A.S.T.M. Designation: D 647).<sup>3</sup> Where no standard design for a mold to produce the desired test specimen has been adopted, the purchaser and the seller should agree as to the mold or molds to be used.

### Molding

3. The press and molding cycle used in molding test specimens shall be agreed upon by the purchaser and the seller.

NOTE.—To permit the use of the same mold for various materials, tolerances in test specimen dimensions of plus or minus 10 per cent for cold-molded, and plus or minus 5 per cent for all other materials shall be permitted, unless otherwise specified.

### CONDITIONING OF TEST SPECIMENS

#### Standard Practice

4. (a) For tests for dielectric strength, volume resistance, power factor, dielectric constant, loss factor, compressive, flexural, impact and tensile strengths, stiffness, distortion under heat, and coefficient of linear thermal expansion, specimens shall be conditioned in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) For all other tests, specimens shall be subjected to no conditioning or to such other particular conditioning as is required.

#### Special Requirements

5. The properties of molded electrical insulating materials to greater or lesser degree are dependent upon the atmospheric conditions of temperature and humidity to which the materials are exposed. In particular, absorption of moisture or exposure to supernormal

temperatures is likely to reduce their value as insulation. Electrical tests after exposure to humid or wet conditions or at elevated temperatures should be made wherever such conditions are likely to be encountered in service. Where insulators may be exposed to either subnormal or supernormal atmospheric conditions and may be subjected to mechanical stresses approaching the maximum they may withstand, materials should be tested for all essential physical properties to such extent as seems advisable following exposure to such conditions. Values obtained for test specimens subjected to special conditioning should be properly identified.

### TEST CONDITIONS

#### Standard Practice

6. (a) Dielectric constant, loss factor, power factor, and volume resistance tests with specimens conditioned (Section 4 (a)) by the Standard Procedure in the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618)<sup>3</sup>, shall be performed at  $25 \pm 1$  C. ( $77 \pm 1.8$  F.) and  $50 \pm 2$  per cent relative humidity. With specimens conditioned by Functional Procedure A in Methods D 618 tests shall be started as soon as possible, preferably within 1 min. after removal of the specimens from the desiccator in which they have cooled to room temperature, and the tests shall be performed under the atmospheric conditions prevailing in the laboratory. Where comparative tests between laboratories are being made, the time interval shall be as agreed upon. If because of lack of equipment, specimens conditioned by the Standard Procedure in Methods D 618 cannot be tested under controlled temperature and humidity, the effect of exposure between conditioning and testing shall be established and reported.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) Compressive, flexural, impact, and tensile strength tests for materials conditioned (Section 4 (a)) by the Standard Procedure in A.S.T.M. Methods D 618 shall be performed at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity. For materials conditioned by Functional Procedure A in Methods D 618 these tests shall be performed at  $25 \pm 2$  C. ( $77 \pm 3.6$  F.) and the humidity prevailing in the laboratory. If, because of lack of equipment, materials conditioned by the Standard Procedure in Methods D 618 cannot be tested under controlled humidity, the effect of exposure between conditioning and testing shall be established and reported.

(c) All other tests are to be performed under atmospheric conditions prevailing in the laboratory or under such other particular conditions as are required.

### Special Requirements

7. Where test specimens are subjected to special conditioning in accordance with Section 5, tests should be performed under like conditions where it is at all practicable to do so, or at least very rapidly if it is necessary to perform them under laboratory atmospheric conditions. For tests of such specimens the use of equipment is recommended, such as is described in the Tentative Specifications for Enclosures for Small Testing Machines for Tests at Subnormal and Supernormal Temperatures of Electrical Insulating Materials and Plastics (A.S.T.M. Designation: D 760)<sup>3</sup> and the Tentative Specifications for Servicing Units for Tests at Subnormal and Supernormal Temperatures of Electrical Insulating Materials and Plastics (A.S.T.M. Designation: D 761).<sup>3</sup>

### ARC RESISTANCE (THERMOSETTING MATERIALS)

#### Procedure

8. Except as specified below in Section 9, arc resistance shall be determined in accordance with the Standard Method of Test for Arc Resistance of Solid Electrical Insulating Materials (A.S.T.M. Designation: D 495).<sup>3</sup>

NOTE.—This method of test serves to classify materials broadly rather than to bring out slight differences between materials of similar composition. Attention is being given to the development of test methods to determine the suitability of molded materials for applications requiring degrees of arc resistance to which some, if not all may be suited.

#### Test Specimen

9. The test specimen shall be  $\frac{1}{8}$  in. in thickness and of any size or shape, provided a flat, or nearly flat, surface of about 0.16 sq. in. in area is available for each test.

### DIELECTRIC STRENGTH

#### Procedure

10. Except as specified in Sections 4(a), 5, 6(a), and 7 and below in Sections 11 to 15, dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149).<sup>3</sup>

#### Electrodes

11. The test electrodes shall consist of cylinders 1 in. in diameter and 1 in. in length, with edges rounded to a radius of  $\frac{1}{8}$  in.

#### Test Specimen

12. The test specimen shall be a disk 4 in. in diameter by  $\frac{1}{4}$  in. in thickness for cold-molded materials and  $\frac{1}{8}$  in. in thickness for all other materials.

### Surrounding Medium

13. It is recommended that cold-molded materials be tested in air, but that all other materials be tested under oil. When arc-over is experienced on specimens tested in air, the specimens shall be tested under oil.

### Test Voltage

14. (a) Either the short-time test or the step-by-step test may be used.

(b) In the short-time test, voltage shall be increased at the rate of 1000 v. per sec.

(c) In the step-by-step test, the voltage shall be increased in increments as indicated in the following table, the voltage being held at each step for 1 min.:

Breakdown Voltage by Short-Time Test, kv.	Increment of Increase of Test Voltage, kv.
25 and under.....	1.0
Over 25 to 50, incl.....	2.0
Over 50 to 100, incl.....	5.0
Over 100.....	10.0

### Number of Tests

15. Five specimens shall be tested by the short-time test and three specimens by the step-by-step test.

### INSULATION AND VOLUME RESISTANCE

#### Procedure

16. Except as specified in Sections 4(a), 5, 6(a), and 7 and below in Sections 17 and 18, insulation and volume resistance shall be determined in accordance with the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257).<sup>3</sup>

#### Test Specimen

17. For resistance measurements emphasizing surface resistance (designated in A.S.T.M. Methods D 257 as insulation resistance measurements) the test specimen shall be a  $\frac{1}{2}$  by  $\frac{1}{2}$  by 5-in. bar pro-

vided with electrodes as shown in Fig. 2 of those methods. For resistance measurements emphasizing volume resistance the test specimen shall be a  $\frac{1}{8}$  by 4-in. disk provided with electrodes as shown in Fig. 4 of A.S.T.M. Methods D 257.

### Test Conditions

18. Test specimens for insulation resistance measurements prior to testing shall be exposed for 96 hr. in air at  $90 \pm 2$  per cent relative humidity and  $35 \pm 1$  C. ( $95 \pm 2$  F.) and shall be tested under the same conditions.

### POWER FACTOR, DIELECTRIC CONSTANT, AND LOSS FACTOR

#### Procedure

19. Except as specified in Sections 4(a), 5, 6(a), and 7 and below in Section 20, power factor, dielectric constant, and loss factor shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

#### Test Specimens

20. The test specimen shall be a disk from 0.100 to 0.150 in. in thickness with the diameter either 2 in.  $\pm$  5 per cent for frequencies of over 1 to 100 megacycles, or 4 in.  $\pm$  5 per cent for frequencies up to and including 1 megacycle. The test specimen shall have smooth surfaces and be free from irregularities (see Section 11 (a) of A.S.T.M. Methods D 150).

### COMPRESSIVE STRENGTH (EXCEPT NON-RIGID PLASTICS)

#### Procedure

21. Except as specified in Sections 4(a), 5, 6(b), and 7 and below in Section 22, compressive strength shall be determined in accordance with the Tentative Method of Test for Compressive Strength

of Plastics (A.S.T.M. Designation: D 695).<sup>3</sup>

### Test Specimen

22. The test specimen shall be a rectangular prism 0.5 by 0.5 by 1.0 in. cut from 0.5 by 0.5 by 5.0-in. bars.

### FLEXURAL STRENGTH (EXCEPT NON-RIGID PLASTICS)

#### Procedure

23. Except as specified in Sections 4(a), 5, 6(b), and 7 and below in Section 24, flexural strength (modulus of rupture) shall be determined in accordance with the Tentative Method of Flexural Test of Plastics (A.S.T.M. Designation: D 790).<sup>3</sup>

### Test Specimen

24. The test specimen shall be 5 by 0.5 by 0.25 in., with 0.25 in. the dimension in the direction in which molding pressure is applied and the beam depth of the specimen as tested.

### IMPACT STRENGTH (EXCEPT NONRIGID PLASTICS)

#### Procedure

25. Except as specified in Sections 4(a), 5, 6(b), and 7 and below in Section 26, resistance to impact shall be determined in accordance with the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup>

### Test Specimen

26. (a) The test specimen for the Charpy test shall conform to the dimensions shown in Fig. 1 but may be notched or unnotched (Note) according to the conditions of service. The notch when used may be milled, or otherwise machined to correct contour, provided the

bottom of the resulting notch is smooth, straight, and free of scratches. The thickness of the specimen as molded, that is, the dimension parallel to the application of molding pressure and lengthwise of the notch may be any thickness of  $\frac{1}{2}$  in. or less agreed upon as representative of the cross-section in which the particular material is to be used.

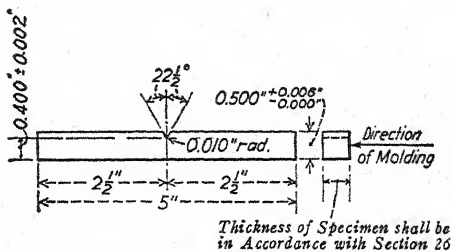


FIG. 1.—Simple Beam (Charpy) Impact Test Specimen.

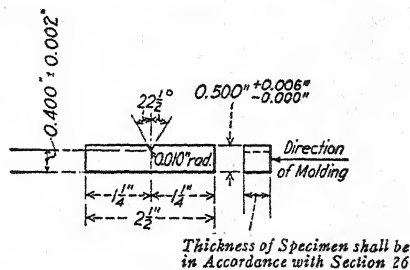


FIG. 2.—Cantilever Beam (Izod) Impact Test Specimen.

NOTE.—The unnotched specimen gives a higher strength than the notched specimen and the ratio of the strength of the unnotched specimen to that of the notched specimen varies with different materials.

(b) The test specimen for the Izod test shall be notched and shall conform to the dimensions shown in Fig. 2. Requirements as to the nature of the notch and the thickness of the specimen shall be the same as for the Charpy test as prescribed in Paragraph (a).

(c) When the individual specimens as molded are less than  $\frac{1}{2}$  in. in thickness, the test specimen shall be a composite

specimen consisting of a number of individual thin specimens aggregating as nearly as possible  $\frac{1}{2}$  in. in thickness. They shall all be accurately aligned with each other and shall be tested edgewise.

#### TENSILE STRENGTH

##### Procedure

27. Except as specified in Sections 4(a), 5, 6(b), and 7, and below in Section 28, the tensile strength of nonrigid plastics shall be determined in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412);<sup>3</sup> the tensile strength of other thermoplastics shall be determined in accordance with the Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638);<sup>3</sup> and the tensile strength of thermosetting or plastic cold-molded materials shall be determined in accordance with the Tentative Method of Test for Tensile Strength of Molded Electrical Insulating Materials (A.S.T.M. Designation: D 651).<sup>3</sup>

##### Test Specimen

28. (a) For nonrigid plastics the test specimen shall be as shown in Fig. 3, Die C of the A.S.T.M. Methods D 412.

(b) For thermoplastic materials other than nonrigid, the test specimen shall be  $\frac{1}{8}$  in. in thickness and the other dimensions shall be as shown for type I specimen in Fig. 1 of A.S.T.M. Method D 638.

(c) For cold-molded materials and thermosetting materials the test specimen shall be molded to the form and dimensions shown in Fig. 2 of A.S.T.M. Method D 651, the use of either the  $\frac{1}{8}$ -in. or the  $\frac{1}{4}$ -in. specimen being permissible (Note). Five specimens are required for a test. Special specimen holders made of steel, as shown in Fig. 1 of A.S.T.M. Method D 651 shall be used.

NOTE.—Values of tensile strength obtained with these two specimens may be unequal and accordingly that specimen should be chosen

which is considered most representative of the material and its intended use.

#### DEFORMATION UNDER LOAD (EXCEPT NONRIGID PLASTICS)

##### Procedure

29. Except as specified in Sections 5 and 7, deformation under load shall be determined in accordance with the Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621).<sup>3</sup>

#### STIFFNESS IN FLEXURE (NONRIGID PLASTICS)

##### Procedure

30. Except as specified in Sections 4 (b), 5, and 7, the stiffness in flexure of nonrigid plastics shall be determined in accordance with the Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (A.S.T.M. Designation: D 747).<sup>3</sup>

#### BRITTLE TEMPERATURE (NONRIGID PLASTICS)

##### Procedure

31. The brittle temperature of nonrigid plastics shall be determined in accordance with the Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746).<sup>3</sup>

#### DISTORTION UNDER HEAT (EXCEPT NONRIGID PLASTICS)

##### Procedure

32. (a) Except as specified in Sections 4 (a) and 5 and below in Section 33, distortion under heat shall be determined in accordance with the Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648).<sup>3</sup>

(b) Unless otherwise specified, the fiber stress shall be 264 psi. for thermoplastic, thermosetting, and cold-molded materials.

**Test Specimen**

33. The test specimen shall be  $\frac{1}{2}$  by 5 in. and of such thickness of  $\frac{1}{2}$  in. or less in the direction in which molding pressure is applied as is agreed to be representative of the cross-section in which the material is to be used. The thickness as molded shall be the width of the beam when under test (Note).

NOTE.—When the specimen is less than  $\frac{1}{4}$  in. in molded thickness, difficulties may be encountered due to tipping over or unsteadiness during test. In such cases the test may be made upon a composite specimen consisting of sufficient individual pieces to aggregate approximately  $\frac{1}{2}$  in. in beam width held together by a rubber band or other light clamp.

**COEFFICIENT OF LINEAR THERMAL  
EXPANSION (EXCEPT NON-  
RIGID PLASTICS)**

**Procedure**

34. Except as specified in Sections 5 and 7, and below in Section 35, coefficient of linear thermal expansion shall be determined in accordance with the Standard Method of Test for Coefficient of Linear Thermal Expansion of Plastics (A.S.T.M. Designation: D 696).<sup>3</sup>

**Test Specimens**

35. Unless otherwise specified, the test specimen shall be at least 4 in. in length.

**FLAMMABILITY (EXCEPT NONRIGID  
PLASTICS)**

**Procedure**

36. Except as specified below in Section 37, flammability under specified conditions of ignition and burning shall be determined in accordance with the Standard Method of Test for Flammability of Plastics Over 0.050 in. in Thickness (A.S.T.M. Designation: D 635).<sup>3</sup>

**Test Specimens**

37. Unless otherwise specified, the test specimens shall be  $\frac{1}{8}$  in. in thickness.

**WATER ABSORPTION****Procedure**

38. Except as specified below in Sections 39 and 40, water absorption shall be determined in accordance with the 24-hr. immersion test described in the Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup>

**Test Specimen**

39. The test specimen shall be a disk 2 in. in diameter by  $\frac{1}{8}$  in. in thickness. Permissible variations in thickness shall be plus or minus 0.012 in. for cold-molded materials and plus or minus 0.007 in. for other materials.

**Conditioning Test Specimens**

40. (a) Specimens of material whose water absorption value would be appreciably affected by temperatures in the neighborhood of 110 C. (230 F.) shall be dried in an oven for 24 hr. at  $50 \pm 3$  C., and cooled in a desiccator.

(b) Specimens of material whose water absorption values have been shown not to be appreciably affected by temperatures up to 110 C. (230 F.) shall be dried in an oven for 1 hr. at 105 to 110 C. (221 to 230 F.).

NOTE.—When data for comparison with values for other plastics are desired, all specimens shall be conditioned in accordance with the procedure described in Paragraph (a).

**CHEMICAL RESISTANCE****Procedure**

41. Resistance to chemical reagents of interest shall be determined in accordance with the Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M. Designation: D 543).<sup>3</sup>

**ACETONE EXTRACTION (PHENOLIC  
MATERIAL)**

**Procedure**

42. The quantity of acetone-soluble matter in molded phenolic materials shall



be determined in accordance with the Standard Method of Test for Acetone Extraction of Phenolic Molded or Laminated Products (A.S.T.M. Designation: D 494).<sup>3</sup>

#### POWDER TESTS

##### Procedure

43. The particle size, apparent density, bulk factor, and pourability of molding powders shall be determined in accordance with the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392).<sup>3</sup>

#### FLOW TEMPERATURE (THERMOPLASTIC MATERIALS)

##### Procedure

44. The flow qualities of thermoplastic materials shall be determined in accordance with the Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (A.S.T.M. Designation: D 569).<sup>3</sup>

#### MOBILITY (THERMOSETTING MATERIALS)

##### Procedure

45. The mobility of thermosetting materials under specified conditions of mold design, applied temperature and pressure shall be determined in accordance with the Tentative Method of Test for Measuring Relative Mobility of Thermosetting Molding Powder (A.S.T.M. Designation: D 731).<sup>3</sup>

#### SHRINKAGE

##### Procedure

46. Initial shrinkage from mold to molded dimensions of either thermoplastic or thermosetting materials shall be determined in accordance with the Standard Method of Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 551).<sup>3</sup>

NOTE.—The purpose of this test is to measure batch-to-batch uniformity in initial shrinkage, and the values obtained may not be representative of those to be expected in other molds.

(See Appendix, p. 535)

## APPENDIX

## UTILITY OF TESTS

A1. The user of a test method should know exactly what it does and does not measure, the precision with which the measurement can be made, the units of value employed, and exactly to what useful purpose or purposes the knowledge gained can be applied. It should be recognized that test values usually are derived from some standard test specimen which has been exposed, intentionally or otherwise, to certain atmospheric conditions, and which has been tested in a certain manner, under prescribed atmospheric conditions. It should not be expected that exactly like unit values, or even that comparable relative values will always be realized, if materials tested are made into utilitarian articles of various sizes, shapes, and cross-sections. The latter may be exposed to detrimental or destructive actions of somewhat different nature and intensity or may be used under atmospheric conditions either more or less favorable to the materials than the test conditions. Allowance should be made for the fact that property values in molded electrical insulation may be affected to variable extents by the design of the article tested, the exact manner of stressing or attack, and the particular atmospheric conditions to which it has been or is exposed. It also may be possible to make some measurements with greater precision than others, and all tests are not applicable to all materials.

A2. The statements which follow are intended to give no more than a convenient indication of the utility of the information gained by performance of the tests listed in Methods D 48. Where complete understanding of a test is desired it should be obtained by reference to the more complete statements of the significance of tests, such as those referred to in Table I of Section 1.

A3. (a) The obvious intent of the following eleven tests is to measure essential electrical properties and mechanical properties, which may be necessary in some electrical insulation:

- Arc Resistance
- Dielectric Constant
- Dielectric Strength
- Insulation Resistance
- Loss Factor
- Power Factor
- Volume Resistance
- Compressive Strength
- Flexural Strength
- Impact Strength
- Tensile Strength

(b) As standardized, commonly performed tests they may be particularly useful in stating specification requirements, since aside from the importance of the properties measured, combinations of values from these tests may be quite characteristic of certain materials and may serve largely to identify them. These are short-time rather than endurance or fatigue tests. Individually and with the limitations in test values to which attention has been called, these tests yield information as to the following:

(1) *Arc Resistance*.—The ability of a material to withstand the discharge across its surface of a specified, at first intermittent and then continuous, high-voltage, low-current arc. Materials may fail through the eventual formation of a conducting path across their surfaces, and must then be judged for arc resistance by their probable ability to withstand some lesser amount of arcing, thought to be the maximum to which they are likely to be exposed.

(2) *Dielectric Constant*.—The ratio of the capacitance of a capacitor in which the material tested is the dielectric to the capacitance of the same capacitor with vacuum (or air) as the dielectric. Test values obtained are a measure of the relative capacity of a material to store electrostatic energy.

(3) *Power Factor*.—The ratio of that part of the current which produces heat in the dielectric to total alternating current which passes through the dielectric.

(4) *Loss Factor*.—The relative tendency of a material to generate heat when used as a dielectric in an electrical field alternating at a specified frequency.

(5) *Dielectric Strength*.—The voltage gradient at which a material fails as a dielectric by electrical breakdown.

(6) *Insulation Resistance*.—The resistance offered by a specimen to the passage of direct current between electrodes attached to it.

(7) *Volume Resistance*.—The resistance offered by a material to the passage of direct current if passage of current through or over its surface layer is prevented.

(8) *Compressive Strength*.—The ability of a material to withstand compressive loads without complete failure.

(9) *Flexural Strength*.—The ability of a material to withstand bending loads without complete failure.

(10) *Impact Strength*.—The ability of a material to withstand a sudden blow without complete failure.



(11) *Tensile Strength*.—The ability of a material to withstand tensile loads without complete failure.

A4. The nature of the following four tests may be more obvious than their utility:

Chemical Resistance

Flammability

Linear Coefficient of Thermal Expansion

Water Absorption

(1) *Chemical Resistance*.—Where electrical insulation may be exposed to chemicals, the chemical resistance test will serve to detect materials attacked seriously by the chemicals used in the test. The test may not detect materials which would deteriorate slowly with prolonged exposure. Since many electrical insulating materials are characterized by inertness to certain chemicals the test may be a useful aid in determining that desired materials are supplied.

(2) *Flammability*.—Fire resistance is desirable in electrical insulation. The flammability test described is a measure of tendency to burn under the specified conditions of ignition and burning but is not a means to determine the likelihood that a material will ignite and burn.

(3) *Linear Coefficient of Thermal Expansion*.—Linear coefficient of thermal expansion is of interest in electrical apparatus where changes in the dimensions of the electrical insulation with changes in temperature may alter its adjustment. The test does not measure changes in dimension which may be caused by prolonged exposure to wet or dry atmospheres with an accompanying gain or loss in moisture or volatile matter. It should be recognized that such changes may occur simultaneously with those due to temperature alone and may overshadow them.

(4) *Water Absorption*.—Water absorption by electrical insulation is of definite interest where insulation may be exposed to humid conditions, since it is likely to affect insulating properties detrimentally. Detrimental effects may vary in different materials and may not be in direct proportion to water absorbed. The test described may be of too short duration to measure maximum possible absorption. The relative tendencies of materials to absorb moisture upon immersion and upon exposure to humid atmospheres may not be exactly the same. The test is primarily a quality control or identification test.

A5. The following nine tests are confined largely to the plastics industry and are less a matter of common engineering knowledge and use:

Acetone Extraction

Brittle Temperature

Deformation Under Load

Distortion Under Heat

Flow Temperature

Mobility

Powder Tests

Shrinkage

Stiffness

(1) *Acetone Extraction*.—Molding material made with phenol-formaldehyde resin becomes largely insoluble in acetone when properly molded. The degree of insolubility depends upon: (a) The adequacy of the heat of the molding operation to harden or react its resin, (b) the insolubility of the resin when reacted, and (c) the presence or absence of other ingredients or substances which remain soluble. A minimum solubility rather than complete insolubility can be achieved, which may indicate either (a) that at least some matter remaining soluble is commonly present in these materials, (b) that none of these resins become completely insoluble, or (c) that ordinarily these resins do not become completely reacted. The quantity of matter which can be extracted from a molding made with phenol-formaldehyde resin, therefore, gives a combined measure of permanently acetone soluble constituents, and of the degree to which its resin has been hardened or reacted, up to the stage where minimum solubility is reached. Extractable matter more volatile than acetone is not evaluated. By themselves acetone extraction values are not a measure of molded quality. If thorough reaction of the resin and freedom from nonvolatile acetone soluble matter is desired low acetone extraction test values give assurance to that effect. Conformity with specified values gives some, but not complete, assurance that a desired molding cycle is being maintained, provided it is known that the desired molding material is being used or *vice versa*. It must be remembered, however, that to measure the solubility of the material all the way through, the moldings tested must be subdivided into small particles in some fashion, since otherwise being little soluble they would be little penetrated by acetone. Acetone extraction values are partly dependent upon how fine or thin these particles are and the heat generated in producing them.

(2) *Brittle Temperature Test*.—The brittle temperature test is a measure of the temperature at which a normally nonrigid plastic reaches a specified degree of brittleness. It is, therefore, some measure of the lowest temperature at which such material is useful.

(3) *Deformation Under Load*.—The deformation under load test gives a measure of the ability of electrical insulation to withstand compression, without yielding and loosening the assembly, in assemblies of conductors and

insulators, which are held together by bolts or rivets. It also gives some measure of hardness. The size and shape of the specimen, the duration of the test, and the temperatures and pressures employed are matters of arbitrary choice. Under other conditions materials may yield to greater or lesser extents.

(4) *Distortion Under Heat.*—The distortion under heat test is a measure of the softening effect of heat upon electrical insulation. The size and shape of the test specimen, the magnitude of the distorting load applied, and the distortion observed, as evidence that a certain temperature is required to soften the material, however, are all matters of arbitrary choice. With other shapes and conditions of loading the effect of heat may be more or less pronounced.

(5) *Flow Temperature, Mobility, and Powder Tests.*—The flow temperature, mobility, and powder tests are applicable to molded electrical insulating materials as supplied for molding. The first two, respectively, are measures of

grades of plasticity in specific thermoplastic and thermosetting molding materials. The third provides measures of the bulk of such materials and of the particle size and pourability of granular materials.

(6) *Shrinkage.*—Molded electrical insulation shrinks to dimensions less than the dimensions of the mold in which it is produced and hence such shrinkage governs the exact dimensions of the molded part produced. The magnitude of the shrinkage depends upon the design of the mold and the molding conditions employed as well as the material. The shrinkage test measures shrinkage in a specified mold which tends to minimize shrinkage and is a measure of batch-to-batch uniformity rather than of shrinkages which should be expected in all molded parts of a given material.

(7) *Stiffness.*—The stiffness test provides a measure of an apparent modulus of elasticity. It gives an index of the ease with which a material may be bent or deformed.

# *Tentative Method of Test for*

## COMPRESSIVE STRENGTH OF ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 649 - 42 T

ISSUED, 1941; REVISED, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers a general procedure for determining the compressive strength of various solid electrical insulating materials. However, modifications are found desirable for different types of such materials and existing methods of the American Society for Testing Materials covering the requirements for test of a given material should be consulted for special details that may apply.

NOTE.—In the case of a material which fails in compression by a shattering fracture, the compressive strength has a very definite value. In the case of materials which do not fail in compression by a shattering fracture, the value obtained for compressive strength is of arbitrary value depending upon the degree of distortion which is regarded as indicating complete failure of the material.

### Apparatus

#### 2. Any universal testing machine may

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1942.

be used provided it is accurate to 1 per cent of the lowest load to be applied. One end of the specimen shall bear upon an accurately centered spherical bearing block located wherever practicable at the top. The metal bearing plates shall be directly in contact with the ends of the specimen.

### Test Specimens

3. Test specimens shall be of the size prescribed for the material being tested. A primary requirement for all test specimens is that the ends shall be flat and perpendicular to the axis. Unless more particularly specified elsewhere the dimensions should conform to the following:

(a) *Molded Materials*.—A cylinder having a height of 1.25 in. (31.8 mm.) and a diameter of 1.125 in. (28.6 mm.).

(b) *Laminated Tubes*.—A section of the tube 1 in. in length.

(c) *Sheet and Plate*.—A 1-in. cube for sheets 1 in. or over in thickness; for sheets less than 1 in. in thickness a pile of sheets 1-in. square with a minimum number of layers to produce a height of at least 1 in.

(d) *Round Rods*.—A section of the rod.

### Conditioning Test Specimens

4. Unless otherwise specified, specimens prior to testing shall be brought to a standard condition in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials.<sup>3</sup>

### Procedure

5. (a) Five conditioned specimens shall be tested at room temperature, or at other temperatures as specified.

(b) The crosshead speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. per min. (1.27 mm. per min.) when the machine is running idle.

(c) Molded materials shall be tested with the load applied on the ends of the specimen.

(d) Laminated tubes shall be tested axially with the load applied perpendicular to the faces or ends of the specimen. Specimens shall also be tested diametrically with the load applied perpendicular to the tangent at the point of application.

(e) Rods shall be tested with the load applied perpendicular to the faces or ends of the specimen.

(f) Sheet and plate materials 1 in. and over in thickness shall be tested both flatwise and edgewise, and cut both crosswise and lengthwise of the sheet. Thinner sheets shall be tested only flatwise and in piles.

### Report

6. The report shall include the following:

(1) The significant dimensions of each specimen in inches measured to the nearest 0.001 in., or in millimeters to the nearest 0.025 mm. and the direction of cutting for sheet and plate specimens. Thickness is the thickness of sheet or plate, or the dimension parallel to the application of molding pressure for molded specimens,

(2) The load on each specimen in pounds or kilograms at the first sign of failure and the direction of application of the load,

(3) The ultimate compressive strength in pounds per square inch, or kilograms per square millimeter, for molded materials and sheets and plates calculated from the measured area before the load is applied and for rods and tubes calculated from the data obtained on the application of the load perpendicular to the face of the specimen,

(4) The rate at which the load was applied, and

(5) Any condition of the specimen or test which has not been standardized for the material.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## *Tentative Methods of*

### CONDITIONING PLASTICS AND ELECTRICAL INSULATING MATERIALS FOR TESTING<sup>1</sup>



A.S.T.M. Designation: D 618 - 46 T

ISSUED, 1941; REVISED, 1944, 1945, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures. for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. Many plastics and electrical insulating materials respond to temperature and relative humidity in a manner that materially affects test results. In order that reliable comparisons may be made of different materials and between different laboratories, it is necessary to standardize the temperature and humidity conditions to which these materials are subjected prior to and during testing. These methods define procedures for conditioning plastics and electrical insulating materials prior to testing, and the conditions under which they shall be tested.

#### Definitions

2. (a) *Standard Laboratory Atmosphere*.—An atmosphere having a relative humidity of  $50 \pm 2$  per cent at a temperature of  $25 \pm 1$  C. ( $77 \pm 1.8$  F.) shall be the Standard Laboratory Atmosphere.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

(b) *Standard Room Temperature*.—An atmosphere of unspecified relative humidity at a temperature in the range of 20 to 30 C. (68 to 86 F.) shall be the Standard Room Temperature.

(c) *Standard Test Temperatures*.—When the effect of temperature on a property is to be determined or reported, the test temperatures shall be chosen from the following:

Test Temperatures	Tolerance, plus or minus
-70 C. (-94 F.)	2.0 C. (3.6 F.)
-55 C. (-67 F.)	2.0 C. (3.6 F.)
-40 C. (-40 F.)	2.0 C. (3.6 F.)
-25 C. (-13 F.)	2.0 C. (3.6 F.)
0 C. (32 F.)	1.0 C. (1.8 F.)
25 C. (77 F.)	1.0 C. (1.8 F.)
50 C. (122 F.)	1.0 C. (1.8 F.)
70 C. (158 F.)	1.0 C. (1.8 F.)
90 C. (194 F.)	1.0 C. (1.8 F.)
105 C. (221 F.)	1.0 C. (1.8 F.)

#### Conditioning Prior to Test

3. (a) *Standard Procedure*.—Test specimens  $\frac{1}{4}$  in. or less in thickness shall be conditioned in the Standard Laboratory Atmosphere (with adequate air circulation) for a minimum period of 40 hr. immediately prior to testing. Test specimens greater than  $\frac{1}{4}$  in. in thickness shall be conditioned in the Standard

Laboratory Atmosphere (with adequate air circulation) for a minimum period of 88 hr. immediately prior to testing. The Standard Procedure shall be used, unless otherwise specified.

NOTE 1.—Standard Procedure is generally satisfactory and recommended for use unless functional procedures are specified.

(b) *Functional Procedure A*.—Test specimens shall be conditioned for a period of 48 hr. in a circulation air oven at a temperature of  $50 \pm 3$  C. ( $122 \pm 5.4$  F.). The specimens shall be removed from the oven and cooled to the Standard Room Temperature in a desiccator over anhydrous calcium chloride (or other suitable desiccant) for a period of at least 16 hr. immediately prior to testing.

NOTE 2.—Functional Procedure A is recommended for use wherever the specific effects of drying are to be determined.

(c) *Functional Procedure B*.—Test specimens shall be conditioned for a period of  $96 \pm 2$  hr. at a temperature of  $35 \pm 2$  C. ( $95 \pm 3.6$  F.) in an atmosphere of 90 per cent relative humidity, the tolerances on humidity being  $\pm 2$  per cent for general purposes and  $\pm 1$  per cent for referee purposes.

NOTE 3.—Functional Procedure B is recommended for use wherever the specific effects of exposure to severe atmospheric moisture are to be determined.

#### Tests at Normal Temperatures

4. (a) Unless otherwise specified, materials conditioned according to the

Standard Procedure shall be tested in the Standard Laboratory Atmosphere.

(b) Unless otherwise specified, materials conditioned according to Functional Procedure A shall be tested at Standard Room Temperature conditions. Not more than  $\frac{1}{2}$  hr. shall elapse between removal of the specimens from the desiccator and the start of the tests.

(c) Unless otherwise specified, materials conditioned according to Functional Procedure B shall be tested in the same atmosphere (Section 3 (c)).

#### Tests at Other Standard Test Temperatures (See Section 2 (c))

5. All materials shall be transferred to the test conditions within  $\frac{1}{2}$  hr., preferably immediately, after completion of the conditioning. The specimens shall be conditioned at the test temperature for not more than 5 hr. prior to tests, and in no case less than the time required to insure thermal equilibrium.

#### Selection of Conditioning Procedure

6. (a) In the case of materials covered by A.S.T.M. specifications, reference should be made thereto to determine the conditioning procedures to be used.

(b) In the case of all other materials, choice between the conditioning procedures should preferably be based on the one which gives the most reproducible test results.

#### Report

7. The report shall state the conditioning procedure used and the atmospheric conditions under which the tests were made.

# *Tentative Method of Test for*

## FLEXURAL STRENGTH OF ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 650 - 42 T

ISSUED, 1941; REVISED, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers a general procedure for determining the flexural strength of various solid electrical insulating materials. However, modifications are found desirable for different types of such materials and existing methods of the American Society for Testing Materials covering the requirements for test of a given material should be consulted for special details that may apply.

NOTE.—For molded materials, the type of mold used to produce test specimens has an effect on the results obtained. Cooperating laboratories should, therefore, standardize mold and testing procedure to obtain concordant results.

### Apparatus

2. Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied. The specimen shall be tested as a simple beam, resting on two parallel supports, and loaded at the center by

means of a pressure piece to apply the load crosswise of the beam. The distance between supports shall be eight times the nominal height of the specimen in test position but not less than  $\frac{1}{2}$  in., or for laminated round rods not more than 4 in. For electrical porcelain, the supports shall be 5 in. apart. Pins may be added to the supports to keep very narrow specimens in a vertical position. The radius of all contact edges shall be  $\frac{1}{8}$  in. (3.18 mm.), except as otherwise specified.

### Test Specimens

3. Test specimens shall be of the size specified for the material being tested, but unless more particularly specified elsewhere, should conform to the following:

(a) *Molded Materials*.—Specimens  $\frac{1}{2}$  by  $\frac{1}{2}$  by 5 in. or of such lesser thickness as is considered representative.

(b) *Sheet and Plate*.—Width of  $\frac{1}{2}$  in., except for specimens over  $\frac{1}{2}$  in. in thickness tested in the flatwise direction in which case the width shall be equal to

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1942.



the thickness of the specimen. The thickness shall be the full thickness of the sheet. The length shall be 1 in. greater than the distance between supports.

(c) *Laminated Round Rods*.—A section of rod 5 in. in length.

(d) *Electrical Porcelain*.—Round rods  $\frac{1}{8}$  in. in diameter by 6 in. in length.

### Conditioning Test Specimens

4. Unless otherwise specified, specimens prior to testing shall be brought to a standard condition in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials.<sup>3</sup>

### Thickness Measurements

5. All thickness measurements shall be made using a machinist's micrometer as described in method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

### Procedure

6. (a) Not less than five specimens (conditioned when necessary) shall be tested at room temperature, or at other temperatures as specified, for each strength value to be determined.

(b) The crosshead speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. per min. (1.27 mm. per min.) when the machine is running idle. Measurements of deflection may be made for very elastic materials.

(c) Molded materials shall be tested with the load applied crosswise of the specimen turned so that its thickness, or dimension parallel to the application of

molding pressure, becomes the width of the beam. Thin specimens less than  $\frac{1}{4}$  in. in thickness which tend to tip over may be assembled to give a composite specimen approximately  $\frac{1}{2}$  in. in width, held together by a rubber band or other light clamp.

(d) Sheet and plate materials shall be tested with specimens turned both flatwise and edgewise, and cut both crosswise and lengthwise of the sheet (Note).

NOTE.—For a description of the testing of sheet and plate materials for flexural strength, reference should be made to Sections 3 and 7 to 10 of the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials.<sup>3</sup>

### Calculation and Report

7. (a) *Calculation*.—The maximum fiber stress in pounds per square inch or kilograms per square centimeter shall be calculated as follows:

For bars of rectangular cross-section:

$$S = \frac{3 Pl}{2 bd^2}$$

For bars of circular section:

$$S = \frac{8 Pl}{\pi d^3}$$

where:

$S$  = maximum fiber stress,

$P$  = breaking load in pounds or kilograms,

$l$  = distance between supports in inches or centimeters,

$b$  = width of the beam as tested in inches or centimeters, and

$d$  = depth of the beam as tested or diameter of specimen in inches or centimeters.

(b) *Report*.—The report shall include the following:

(1) The depth and width (or diameter determined from at least two measurements 90 deg. apart) of each specimen measured by a micrometer (Section 5)

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



expressed in inches to the nearest 0.001 in. or in centimeters to the nearest 0.025 mm.,

(2) The direction of cutting and loading specimens of sheet and plate materials (see Note, Section 6 (d)),

(3) The breaking load in pounds or kilograms,

(4) The maximum fiber stress in pounds per square inch or kilograms per square centimeter calculated in accordance with Paragraph (a),

(5) The rate at which the load was applied,

(6) The maximum deflection at the center, in inches or millimeters if measured,

(7) A description of the fracture if significant,

(8) Any condition of the specimen which has not been standardized for the material, and

(9) Any other information required under a specific material test method.

## *Tentative Methods of Test for*

### IMPACT RESISTANCE OF PLASTICS AND ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 256 - 43 T

ISSUED, 1941; REVISED, 1943.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. (a) These methods of test are intended to determine the relative susceptibility to fracture by shock of plastic materials and electrical insulating materials as indicated by the energy expended by a standard pendulum type impact machine in breaking a standard specimen in one blow.

(b) There are two types of pendulum impact machines and related methods of test, which use different specimens and differ in the method of holding and striking the specimen. Each specimen and method has characteristics that may dictate its use. Results by the different methods cannot be directly compared, because impact values determined by the two methods may be numerically different.

(c) *Method A* is the cantilever beam or Izod type test in which the specimen is held as a cantilever beam (usually vertical) and is broken by a blow delivered at a fixed distance from the edge of the specimen clamp. The test requires a notched specimen in all cases. The notch is intended to produce a standard degree of stress concentration.

(d) *Method B* is the simple beam or Charpy type test in which the specimen is supported as a simple beam (usually horizontal) and is broken by a blow delivered midway between the supports. In this test the specimen may be either plain or notched, as required by the characteristics of the material tested.

#### *Method A. Cantilever Beam (Izod Type) Test*

##### Apparatus

2. (a) The machine for method A shall be of the pendulum type as shown in Fig. 1, of rigid construction, and accurate to 0.01 ft-lb. for readings of less than 1 ft-lb. and to 1 per cent for higher values. Accurate correction shall be made for friction and windage losses.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, September 24, 1943.

Prior to their present publication as tentative, these methods were published as tentative from 1926 to 1938, being revised in 1928, 1932, 1934, and 1938. They were adopted in 1938, published as standard from 1938 to 1942, but withdrawn as standard in 1942.

(b) The dimensions of the machine shall be such that the center of percussion of the striker (Note) is at the point of impact, that is, the center of the striking edge.

NOTE.—The distance from the axis of support to the center of percussion,  $l$ , may be determined experimentally from the period of oscillation of the pendulum through a small angle by means of the expression  $l = 0.81 P^2$ , where  $P$  is the time in seconds of a complete swing (to and fro).

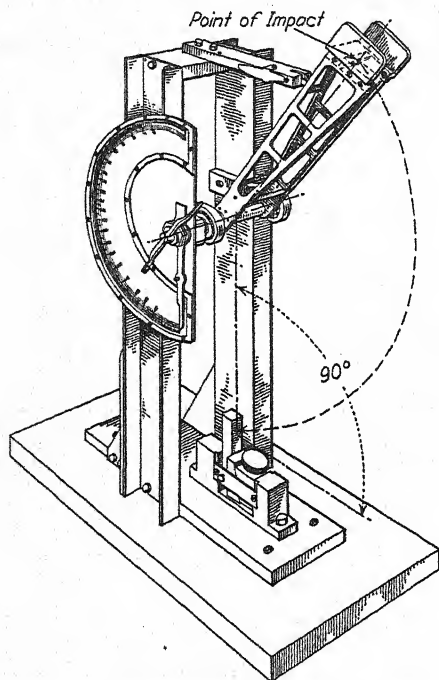


FIG. 1.—Cantilever Beam (Izod Type) Impact Machine.

(c) The pendulum shall be released from such a position that the linear velocity of the center of the striking edge at the instant of impact shall be approximately 11 ft. per sec., which corresponds to an initial elevation of this point of 2 ft.

(d) The striking edge of the pendulum shall be a circular cylindrical surface of

$\frac{1}{32}$ -in. radius, with its axis horizontal. The cylindrical surface shall be, when the pendulum is hanging free, tangent to the specimen in a line 0.866 in. above the top surface of the vise. The pendulum above the cylindrical portion of the striking edge shall be recessed or inclined at a suitable angle so that there is no chance of its coming in contact with the specimen during the break.

(e) Means shall be provided for clamping the specimen rigidly in position with the edges of the supporting surfaces at 90-deg. angles.

(f) Means shall be provided for determining the impact value of the specimen, which is the energy expended by the machine in breaking the specimen. This value is equal to the difference between the energy in the pendulum blow and the energy remaining in the pendulum after breaking the specimen, after suitable correction has been made for windage and friction.

### Test Specimen

3. (a) The test specimen shall conform to the dimensions shown in Fig. 2, except

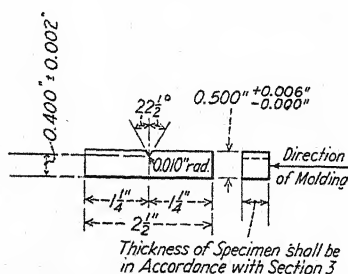


FIG. 2.—Cantilever Beam (Izod Type) Impact Test Specimen.

as modified in accordance with Paragraphs (b), (c), and (d). To insure duplication of the correct contour of the specified notch, all specimens shall be notched with a special formed milling cutter or other equivalent means and in

such manner that the bottom of the notch is smooth, straight, and free of scratches.

(b) For molded material (Note 1) the specimen shall be  $\frac{1}{2}$  in. by any dimension of  $\frac{1}{2}$  in. or less agreed upon as representative of the cross-section in which the particular material is to be used. For all specimens having one dimension less than  $\frac{1}{2}$  in., the notch shall be cut in the narrower side. For all compression molded specimens, the notch shall be in the side parallel to the direction of application of the molding pressure (Note 2).

NOTE 1.—The type of mold used to produce test specimens has an effect on the results obtained. Cooperating laboratories should, therefore, agree upon standard molds conforming to the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (A.S.T.M. Designation: D 647) of the American Society for Testing Materials,<sup>3</sup> and upon molding procedure, to obtain concordant results.

NOTE 2.—The impact strength may be different if the notch is perpendicular to the direction of molding instead of parallel.

(c) For sheet material the specimens shall be cut from the sheet in both the lengthwise and crosswise directions, unless otherwise specified (Note 3). The thickness shall be the thickness of the sheet except that it shall not exceed  $\frac{1}{2}$  in. Sheet material thicker than  $\frac{1}{2}$  in. shall be machined down to  $\frac{1}{2}$  in. Such material  $\frac{1}{2}$  in. or thicker than  $\frac{1}{2}$  in. may be tested either edgewise or flatwise, as specified. When specimens are tested flatwise, the notch shall be made on the machined surface if the specimen is machined on one side only. When the specimen is cut from a thick sheet, notation shall be made from what portion of the thickness of the sheet the specimen is cut, for example, center, top surface, or bottom surface.

NOTE 3.—In referring to the cutting of the specimens of laminated sheet materials and the application of the load, the following descriptions of terms apply:

*Flatwise.*—Load applied to the flat side of the original sheet or plate.

*Edgewise.*—Load applied to the edge of the original sheet or plate.

*Lengthwise.*—In the direction of the length of the sheet.

*Crosswise.*—In the direction at right angles to the direction of the length of the sheet.

When the sheet has the same length and width, one dimension shall arbitrarily be designated as the "A" direction and the other as the "B" direction.

(d) When the individual specimen is less than  $\frac{1}{2}$  in. in thickness, the test specimen may be a composite specimen consisting of a number of individual thin pieces aggregating as nearly as possible  $\frac{1}{2}$  in. in thickness. The individual members of the test specimen shall all be accurately aligned with each other and shall be tested edgewise. Single specimens less than  $\frac{1}{2}$  in. in thickness may be used, provided the width is sufficient for firm, accurate clamping and the impact value of the material is sufficiently high to be accurately determined by a machine of the capacity used.

#### Conditioning Test Specimens

4. Specimens shall be preconditioned and tested in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

#### Procedure

5. (a) At least five individual determinations of impact value shall be made under the conditions prescribed in Section 4.

(b) The test specimen shall be rigidly clamped with the center line of the notch on the level of the top of the clamping surface (Note) and the blow shall be struck on the notched side.

(c) When a composite specimen is used, the individual members shall be

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

held closely in contact and accurately aligned with each other when clamped (Note).

NOTE—It is recommended that a jig or template be used to locate the specimen in the jaws, as specified.

### Report

6. The report shall include the following:

(1) A statement indicating the size of specimen, the method of test and type of preconditioning used, and, for sheet materials, the direction of testing and whether the specimens were cut lengthwise or crosswise from the sheet,

(2) The value of energy expended in breaking each individual specimen expressed in foot-pounds per inch of notch, determined by dividing the energy in foot-pounds expended in the individual test by the actual dimension in inches along the notch of the specimen broken in each test, and whether a single or a composite specimen was used and also the direction of cutting the specimen, and

(3) The average of the values given in Item (2), the average thickness of the individual specimen, and the number of such specimens broken in each operation of the machine.

### *Method B. Simple Beam (Charpy Type) Test*

#### Apparatus

7. (a) The machine for Method B shall be of the pendulum type as shown in Fig. 3, of rigid construction, and accurate to 0.01 ft.-lb. for readings of less than 1 ft.-lb. and to 1 per cent for higher values. Accurate correction shall be made for friction and windage losses.

(b) The dimensions of the machine shall be such that the center of percussion of the striker (Note, Section 2) is at

the point of impact, that is, the center of the striking edge.

(c) The pendulum shall be released from such a position that the linear velocity of the center of the striking edge (center of percussion) at the instant of impact shall be approximately 11 ft. per sec., which corresponds to an initial elevation of this point of 2 ft.

(d) The striking edge of the pendulum shall be tapered to have an included

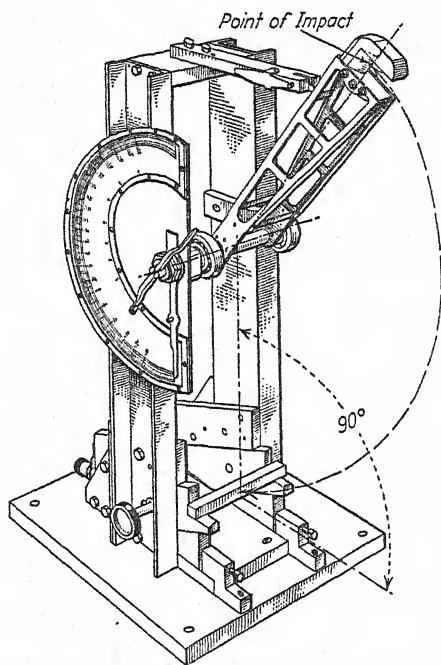


FIG. 3.—Simple Beam (Charpy Type) Impact Machine.

angle of 45 deg. and shall be rounded to a radius of 0.125 in. It shall be so aligned that in the case of rectangular specimens it will make contact across the full width of the specimen.

(e) The test specimen shall be supported against two rigid blocks in such a position that its center of gravity shall lie on a tangent to the arc of travel of the center of percussion of the pendulum drawn at the position of impact. The

edges of the blocks shall be rounded to a radius of 0.125 in. and the points of support shall be 4 in. apart.

(f) Means shall be provided for determining the impact value of the specimen, which is the energy expended by the machine in breaking the specimen. This value is equal to the difference between the energy in the pendulum blow and the energy remaining in the pendulum after breaking the specimen, after suitable correction has been made for friction and windage.

### Test Specimen

8. (a) For materials other than ceramic materials, the test specimen shall conform to the dimensions shown in Fig. 4,

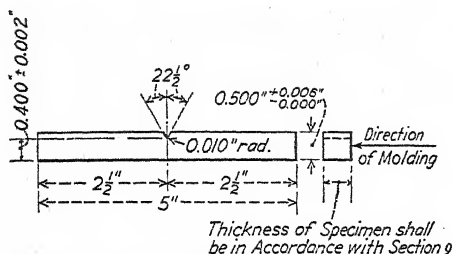


FIG. 4.—Simple Beam (Charpy Type) Impact Test Specimen.

except as modified in accordance with Paragraphs (b), (c), and (d). The notch, when used, shall be milled with a special formed milling cutter or machined by other equivalent means to insure duplication of correct contour and in such manner that the bottom of the notch is smooth, straight, and free of scratches.

(b) For molded material (Note 1, Section 3), the specimen shall be  $\frac{1}{2}$  in. by any dimension of  $\frac{1}{2}$  in. or less agreed upon as representative of the cross-section in which the particular material is to be used. For all specimens having one dimension less than  $\frac{1}{2}$  in., the notch, when used, shall be cut in the narrower side. For all compression molded specimens,

the notch shall be in the side parallel to the direction of application of the molding pressure.

(c) For sheet material, the thickness shall be the thickness of the sheet, except that it shall not exceed  $\frac{1}{2}$  in. Sheet material thicker than  $\frac{1}{2}$  in. shall be machined down to  $\frac{1}{2}$  in. Sheet material thicker than  $\frac{1}{2}$  in. may be tested either edgewise or flatwise, as specified, and may be cut from the sheet either lengthwise or crosswise, as specified (Note 3, Section 3). When specimens are tested flatwise, the notch, when used, shall be made in the original surface.

(d) When the individual specimen is less than  $\frac{1}{2}$  in. in thickness, the test specimen may be a composite specimen consisting of a number of individual thin pieces aggregating as nearly as possible  $\frac{1}{2}$  in. in thickness. The individual members of the test specimen shall all be accurately aligned with each other and shall be tested edgewise. Single specimens less than  $\frac{1}{2}$  in. in thickness may be used, provided the width is sufficient to insure stability during the test and the impact value of the material is sufficiently high to be accurately determined by a machine of the capacity used.

(e) *Ceramic Material.*—For ceramic materials, the specimen shall be an unnotched cylinder 6 in. in length. The diameter shall be 0.5 in., 0.75 in., or 1.125 in., whichever value is comparable to that of the finished product.

### Conditioning Test Specimens

9. Except for ceramic materials, specimens shall be preconditioned and tested in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

### Procedure

10. (a) At least five individual determinations of impact value shall be made

under the conditions prescribed in Section 9.

(b) The test specimen shall be supported against the steel blocks so that the blow will be struck at the center of the specimen, and on the side opposite the notch for notched specimens.

(c) When a composite specimen is used, the individual members shall be closely in contact and accurately aligned with each other.

#### Report

11. The report shall include the following:

(1) A statement indicating the size of specimen, the method of test and type of preconditioning used, and, for sheet materials, the direction of testing and whether the specimens were cut lengthwise or crosswise from the sheet,

(2) For ceramic materials, the energy expended in breaking each individual specimen expressed directly, and for other materials, the value of energy expended in breaking each individual specimen expressed in foot-pounds per inch of notch, or per inch of width of the face of the specimen against which the hammer strikes, determined by dividing the energy in foot-pounds expended in the individual test by the actual dimension in inches along the notch or face of the specimen broken in each test; also, whether a single or a composite specimen was used, and

(3) The average of the values given in Item (2), the average thickness or diameter of the individual specimen, and the number of such specimens broken in each operation of the machine.



# *Tentative Method of Test for*

## TENSILE STRENGTH OF MOLDED ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 651 - 42 T

ISSUED, 1941; REVISED, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers the general procedure for determining the tensile strength of various molded electrical insulating materials. However, modifications in the procedure may be desirable for different types of such materials and existing methods of the American Society for Testing Materials covering the requirements for testing a given material should be consulted for special details that may apply.

### Apparatus

2. Any universal testing machine may be used, provided it is accurate to 1 per cent of the lowest load to be applied. Special specimen holders made of steel, as shown in Fig. 1, shall be used for such test specimens as are of a shape to fit them. The specimen holders shall be attached to the heads of the testing machine by links held so that the pull is central to avoid any transverse

strain. Equally suitable holders, suitably mounted, shall be used for specimens of other shapes.

### Test Specimen

3. Unless otherwise required, the test specimen for hot-molded and plastic cold-molded materials shall be molded to the form and dimensions shown in Fig. 2, the use of either the  $\frac{1}{8}$ -in. or the  $\frac{1}{4}$ -in. specimen being permissible. Values for tensile strength obtained with these two specimens may be unequal and in such cases the specimen which is considered most representative of the material and its intended use shall be tested.

### Mold

4. The specimens shall be molded in molds conforming to Fig. 3 of the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (A.S.T.M. Designation: D 647) of the American Society for Testing Materials.<sup>3</sup> The pressure and other molding conditions used in molding test specimens shall be agreed upon by the seller and the purchaser.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1942.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



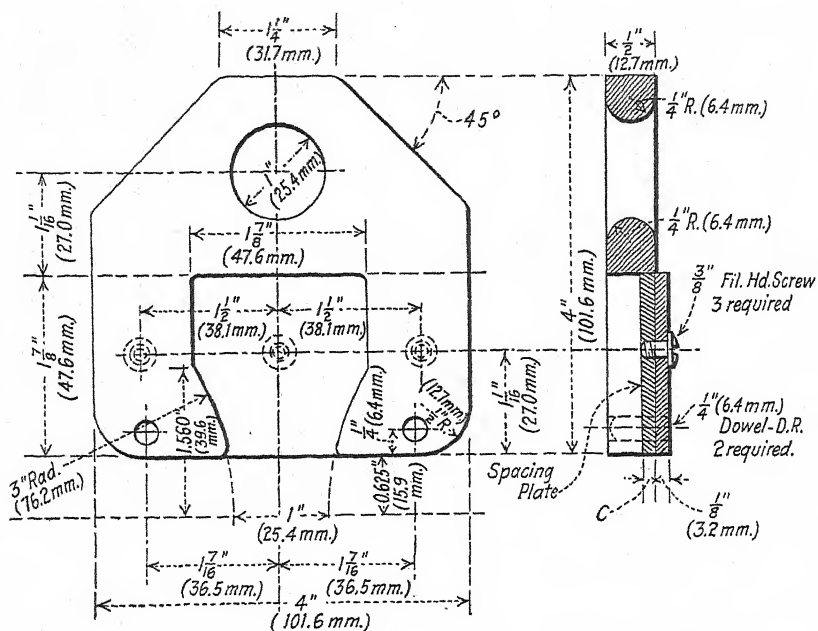


FIG. 1.—Specimen Holder.

	Thickness of Spacing Plate, C
For $\frac{1}{8}$ -in. specimen.....	0.125 in. (3.2 mm.)
For $\frac{1}{4}$ -in. specimen.....	0.062 in. (1.6 mm.)

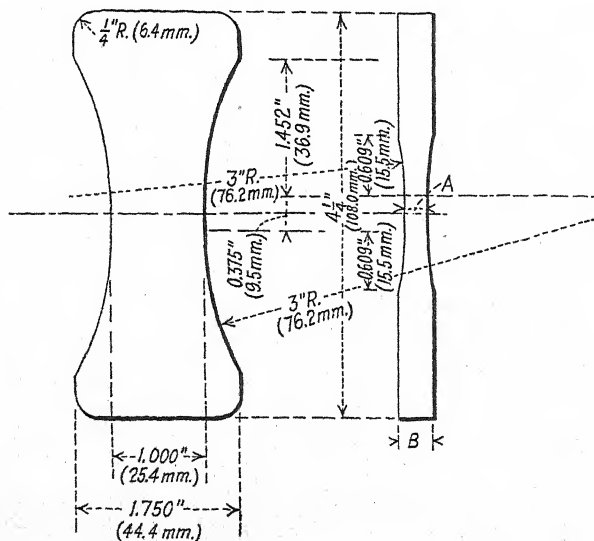


FIG. 2.—Tension Test Specimen.

	Dimension A	Dimension B
For $\frac{1}{8}$ -in. specimen.....	0.125 in. (3.2 mm.)	0.250 in. (6.3 mm.)
For $\frac{1}{4}$ -in. specimen.....	0.250 in. (6.3 mm.)	0.375 in. (9.5 mm.)

NOTE.—To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds.....	±5 per cent
For cold-molded compounds.....	±10 per cent

### Conditioning Test Specimens

5. Unless otherwise specified, all specimens prior to testing shall be conditioned in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials.<sup>3</sup>

### Test Conditions<sup>4</sup>

6. Tensile strength tests for materials conditioned (Section 5) by the Standard Procedure in A.S.T.M. Methods D 618 shall be performed at  $25 \pm 1$  C. ( $77 \pm 1.8$  F.) and  $50 \pm 2$  per cent relative humidity. For materials conditioned by Functional Procedure A in Methods D 618, these tests shall be performed at  $25 \pm 2$  C. ( $77 \pm 3.6$  F.) and the humidity prevailing in the laboratory. If, because of lack of equipment, materials conditioned by the Standard Procedure in Methods D 618 cannot be tested under controlled humidity, the effect of exposure between conditioning the testing shall be established and reported.

### Procedure

7. Five conditioned specimens shall be tested for each type of molded mate-

rial, each specimen being properly aligned in the holders and broken. The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. (1.27 mm. per min.) with the machine running idle.

### Report

8. The report shall include the following:

(1) The breaking load of each specimen in pounds or kilograms,

(2) The thickness and width of each specimen in inches or millimeters as measured by a micrometer at the center of the specimen, that is, the point of minimum section.

(3) The ultimate tensile strength in pounds per square inch or in kilograms per square millimeter of each specimen, calculated from the minimum area measured at the center of the test specimen before the load is applied,

(4) The character of the material tested, with a description of the fracture and its location with respect to the middle point of the specimen,

(5) The speed in inches or millimeters per minute at which the head of the testing machine traveled during the test, and

(6) The elongation in inches or millimeters if determined.

<sup>4</sup>This section editorially revised in October, 1945, and January, 1947.

# *Tentative Specifications for* LAMINATED THERMOSETTING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 709 - 46 T

ISSUED, 1943; REVISED, 1944, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover laminated thermosetting materials consisting of two or more plies or layers of sheet fillers bonded by a thermosetting type of synthetic resin. Examples of such sheet fillers are cellulose paper, asbestos paper, cotton fabric, asbestos fabric, glass fabric, wood veneer, etc.

NOTE 1.—These specifications do not cover special grades of material for radio or similar critical electrical applications. These grades are covered by the Tentative Specifications for Phenolic Laminated Sheet for Radio Applications (A.S.T.M. Designation: D 467)<sup>3</sup> and the Tentative Specifications for Round Phenolic Laminated Tubing for Radio Applications (A.S.T.M. Designation: D 616)<sup>3</sup> of the American Society for Testing Materials.

NOTE 2.—The properties included in these specifications are those required to identify the types and grades of laminated thermosetting

materials covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types and Grades

2. *Types and Grades.*—The types and grades of laminated materials covered by these specifications are classified in accordance with the sheet filler employed in their manufacture, and the electrical, mechanical, and heat-resisting characteristics of the finished products (Note 3). Three types and twelve grades of laminated thermosetting material are covered, as follows:

*Type I.* Cellulose paper base:

- Grade X. Mechanical,
- Grade P. Mechanical; punching stock,
- Grade XX. Electrical and mechanical,
- Grade XXP. Electrical and mechanical; punching stock,
- Grade XXX. Electrical and high humidity, and
- Grade XXXP. Electrical and high humidity; punching stock.

*Type II.* Cellulose fabric base:

- Grade C. Mechanical,

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the joint jurisdiction of A.S.T.M. Committees D-9 on Electrical Insulating Materials and D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Grade CE. Mechanical and electrical,

Grade L. Mechanical; fine machining, and

Grade LE. Mechanical and electrical; fine machining.

*Type III. Asbestos base:*

Grade A. Asbestos-paper base; heat resistant, and

Grade AA. Asbestos-fabric base; mechanical and heat resistant.

NOTE 3.—Further descriptive information regarding these various types and grades of laminated thermosetting materials is given in the Appendix.

**Forms**

3. *Forms.*—Laminated thermosetting materials are available in four forms: sheets, tubes (Note 4), rods (Note 5), and molded shapes (Note 6) as indicated in Table I. These specifications cover

TABLE I.—AVAILABLE TYPES, GRADES, AND FORMS OF LAMINATED THERMOSETTING MATERIALS.

Type	Grade	Forms				
		Sheets	Round Tubes		Round Rods	
			Rolled	Molded	Molded	Machined
I.....	X	a	a	a		b
	P	a				
	XX	a	a	a	a	b
	XXP	a				
	XXX	a		b	a	b
II.....	C	a	a		a	b
	CE, L	a	a	a	a	b
	LE	a	a	a	a	b
III.....	A, AA	a	b	b	a	b

<sup>a</sup> Materials covered by these specifications.

<sup>b</sup> Detailed requirements on these materials are not yet available.

<sup>c</sup> Materials not covered by these specifications.

the material in three forms: sheets, tubes of two classes (rolled and molded), and molded rods. The classes of tubes and of molded rods desired shall be specified by the purchaser in the contract or order.

NOTE 4.—Tubes are made of laminations of fibrous sheet impregnated material, rolled upon mandrels under tension or between heated pressure rolls, or both. They are of two classes,

rolled and molded. Rolled tubes are oven-baked after rolling on the mandrels. Molded tubes are cured in molds under heat and pressure.

NOTE 5.—Molded rods are composed of laminations of impregnated sheet material molded in cylindrical molds under heat and pressure, and then ground to size. Molded rods are of two classes made by winding the impregnated sheet convolutely before molding or by forming strips in the molding operation.

Machined rods, manufactured from certain grades of sheet material, are not covered by these specifications. In rods machined from sheets, the laminations are parallel chords of a circular cross-section. In general, the properties of these rods conform to the grade of sheet stock from which they are cut. This type of rod is low in flexural strength when stress is applied perpendicular to the lamination.

NOTE 6.—Molded shapes are composed of impregnated sheet materials cut into various sizes and shapes to fit the contours of a mold, and molded under heat and pressure. In special cases some macerated material is used in combination with impregnated sheet materials, depending upon the design of the piece. The requirements of these specifications, particularly with regard to mechanical properties, cannot be considered as applying to molded shapes since such properties will depend to a considerable extent upon the design of the piece.

**General Requirements**

4. (a) *Material and Workmanship.*—The laminated material shall be uniform in quality (Note 7). It shall be free from blisters, wrinkles, or cracks and reasonably free from other small defects such as scratches, dents, heat marks, etc., as defined in the Standard Descriptive Nomenclature of Objects Made from Plastics (A.S.T.M. Designation: D 675) of the American Society for Testing Materials.<sup>3</sup> Tubes with wall thickness greater than  $\frac{1}{2}$  in. and molded rods having diameters greater than 1 in. may show checks or cracks between the laminations on machined or sawed edges.

NOTE 7.—In general, most of the grades can be drilled, tapped, sawed, and machined. Grades X, P, and A, are not recommended for drilling and tapping parallel with laminations. Grades XXX, CE, and LE are best suited to these operations.

(b) *Finish and Color.*—Requirements for finish (Note 8) and color (Note 9) shall be as specified by the purchaser in the contract or order.

NOTE 8.—The various forms and grades of laminated thermosetting material are available in the following finishes:

Form	Grade	Finish <sup>a</sup>
Sheets.....	XX, XXX, CE, LE..... X, C, L, A, AA, P, XXP, XXXP	Semigloss or Polished Semigloss only Semigloss or Dull
Round tubing and molded rods.....	All.....	Ground, Buffed, or Varnished
Rectangular and Square Molded Tubes.....	All.....	Semigloss, Ground, or Varnished

<sup>a</sup> *Ground finish* is applied by a fine grinding wheel or belt, is free from any pronounced scratches, and suitable for a majority of applications.

*Buffed finish* is somewhat more glossy than the ground finish, and is obtained by buffing the tube or rod following grinding using a touch of shellac or other polishing compound on the buffer.

*Varnished finish* is sometimes applied to tubes or rods for special decorative, special electrical, or chemical-resistant applications. It is obtained by coating the tube or rod one or more times with a varnish or lacquer and air drying or baking. Because the varnish finish requires operations which may change greatly the physical and electrical properties, such finished tubes or rods are not covered by these specifications.

NOTE 9.—The various types and grades of laminated thermosetting material are available in the following colors:

Type	Grade	Color <sup>a</sup>
I.....	X, P.....	Natural, Black, or Chocolate
	XX, XXX.....	Natural, Black
	XXP, XXX.....	Natural, Black, or Chocolate
	XXXP.....	Natural
II.....	C, CE, L, LE.....	Natural, Black
III.....	A.....	Natural, or Gray-Black
	AA.....	Natural

<sup>a</sup> *Natural color* is produced by the natural undyed paper or fabric and resin used. This natural color shall be free from streaks or stains and substantially uniform, except for grades XXP and XXXP which may be mottled in appearance.

*Black color* is produced by natural undyed paper or fabric and black dyed resin. Sheets and tubes shall have substantially uniform black surfaces and body. A slight grayish black color on sawed, sanded or machined surfaces is permissible, or in the case of tubes where layers may be partially cut through in grinding the tube to diameter. For fabric and asbestos paper base materials, a decided grayish black color is permissible on sawed, sanded, machined or ground surfaces and edges.

*Chocolate colored sheets* have a uniform dark brown or chocolate color surface with natural core.

(c) *Warp or Twist.*—The warp or twist shall not exceed the values prescribed in Table II.

TABLE II.—PERMISSIBLE WARP OR TWIST.

Form	Thickness or Outside Diameter, in.	Permissible Warp or Twist (on Basis of 36-in. Dimension), max., per cent <sup>a</sup>
Sheets.....	{ Over $\frac{1}{8}$ to $\frac{1}{4}$ , incl....	1
	{ Over $\frac{1}{4}$ to $\frac{3}{4}$ , incl....	0.5
	{ Over $\frac{3}{4}$ .....	0.25
Tubes and molded rods.....	{ $\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	2
	{ $\frac{1}{4}$ to $\frac{3}{4}$ , incl.....	1
	{ Over $\frac{3}{4}$ .....	0.5

<sup>a</sup> In the case of warp this percentage is stated in terms of the lateral dimensions (length or width); in the case of twist which only applies to sheet forms the percentage is stated in terms of the dimensions from one corner to the opposite corner.

(d) *Punching Properties.*—The punching quality of the various grades (Note 10) shall be subject to agreement by the manufacturer and the purchaser.

NOTE 10.—The punching properties of the fabric base grades and of the paper base grades classed as punching stock are somewhat better than those of the other paper base grades. All grades can be punched in thin thicknesses under suitable conditions.

#### Detail Requirements for Sheet

5. Sheet material shall conform to the requirements as to physical and electrical properties prescribed in Tables III, IV, and V.

#### Detail Requirements for Tubes

6. Tubes shall conform to the requirements as to physical and electrical properties prescribed in Table VI.

#### Detail Requirements for Molded Rods

7. Molded rods shall conform to the requirements as to physical and electrical properties prescribed in Tables VII and VIII.

#### Sheet Sizes and Permissible Variations in Dimensions

8. (a) *Length and Width.*—The length and width of sheets may vary 1 in. over

TABLE III.—DETAIL REQUIREMENTS FOR SHEETS IN THICKNESSES OF  $\frac{1}{8}$  TO 1 IN., INCLUSIVE.<sup>a</sup>

NOTE.—All values are based on tests at room temperature of approximately 25 C. (77 F.).

Type	Grade	Tensile Strength, min., psi.	Flexural Strength, min., psi.	Compressive Strength, Flatwise, min., psi.	Impact Strength, (Izod), min., ft.-lb. per in. of width		Power Factor at 1,000,000 cycles, max.	Dielectric Constant at 1,000,000 cycles, max.	Loss Factor at 1,000,000 cycles, max.
					Flatwise <sup>b</sup>	Edge-wise			
I.....	X.....	10 000	20 000	none	1.3	0.50	none	none	none
	P.....	6 000	11 000	none	...	0.50	none	none	none
	XX.....	8 000	12 000	none	1.0	0.40	0.045	5.5	0.25
	XXX.....	6 000	12 000	none	...	0.40	0.045	5.5	0.25
	XXX.....	6 000	12 000	none	0.80	0.35	0.035	5.2	0.18
	XXXXP.....	5 000	12 000	none	...	0.30	0.030	5.2	0.16
II.....	C.....	7 500	16 000	35 000	3.2	2.0	none	none	none
	CE.....	6 500	13 000	34 000	2.3	1.3	0.065	6.0	0.40
	L.....	7 000	15 000	30 000	2.5	1.2	none	none	none
	LE.....	6 500	15 000	33 000	1.8	1.0	0.055	5.5	0.30
III.....	A.....	5 000	10 000	30 000	1.8	0.80	none	none	none
	AA.....	8 000	16 000	35 000	3.5	3.0	none	none	none

<sup>a</sup> Minimum requirements for mechanical properties for sheets in thicknesses from over 1 to 2 in. inclusive, shall be 10 per cent lower than those given in this table.<sup>b</sup> Flatwise impact tests are applicable only to sheets having a thickness of  $\frac{1}{8}$  in. or over, and machined to  $\frac{1}{8}$  in. in thickness. No flatwise impact values are given for grades P, XXX, or XXXP as these grades are not available in thicknesses exceeding  $\frac{1}{8}$  in.

or under the manufacturer's standard size (Note 11). When smaller size sheets are cut from standard size sheets, the permissible variations from the specified length or width shall be within the requirements prescribed in Table IX.

TABLE IV.—DIELECTRIC STRENGTH REQUIREMENTS FOR SHEETS.<sup>a</sup>

Thickness, in.	Dielectric Strength Perpendicular to Laminations, min., v. per mil			
	Type I		Type II	
	Grades X, P, XX, XXX, XXXX, and XXXXP		Grades CE <sup>b</sup> and LE	
	Short-Time Test	Step-by-Step Test	Short-Time Test	Step-by-Step Test
$\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	700	450	560	350
Over $\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	500	300	400	240
Over $\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	360	220	290	170
Over $\frac{1}{2}$ to $\frac{3}{4}$ , incl.....	250	150	200	120
Over $\frac{3}{4}$ to 1, incl.....	180	110	145	85
Over $\frac{1}{2}$ to $\frac{3}{4}$ , incl.....	145	90	120	70
Over $\frac{3}{4}$ to 1, incl.....	130	80	105	60
Over 1 to 2, incl.....	90	55	75	40

<sup>a</sup> There are no dielectric strength requirements for grades C, L, A, and AA since these grades are manufactured to meet mechanical requirements and are only suitable for low-voltage electrical applications.<sup>b</sup> For grade CE, short-time and step-by-step dielectric strength test requirements do not apply to sheets in thicknesses less than  $\frac{1}{8}$  in.

NOTE 11.—In general, manufacturer's standard widths are between 36 and 48 in.

(b) *Thickness.*—The permissible variations from the standard thicknesses (Note 12) of the various grades of sheets shall be within the requirements prescribed in Table X. At least 90 per cent of the area of the sheet shall be within the variations prescribed in Table X and at no point shall the thickness as measured vary from the nominal by a value greater than 125 per cent of the specified variation.

NOTE 12.—Sheets are available in the following thicknesses:

Type	Grade	Thicknesses Available, in.	
		Min.	Max.
I.....	X, XX.....	0.010	2
	P.....	0.010	$\frac{1}{8}$
	XXX, XXXP.....	0.015	$\frac{1}{4}$
	XXXX.....	0.015	2
II.....	C.....	$\frac{1}{8}$	10
	CE.....	$\frac{1}{8}$	2
	L.....	0.010	2
	LE.....	0.015	2
III.....	A.....	0.025	2
	AA.....	$\frac{1}{8}$	2

### Tube Sizes and Permissible Variations in Dimensions

9. (a) *Length*.—The length of rolled or molded tubes may vary within plus or minus 1 in. from the manufacturer's

NOTE 13.—Tubes are available in manufacturers' lengths which vary from 18 to 24 in. in small outside diameters and from 30 to 48 in. in large diameters. In a number of diameters of certain grades, longer lengths are available.

TABLE V.—WATER ABSORPTION REQUIREMENTS FOR SHEETS.

Thickness, in. <sup>a</sup>	Water Absorption After 24-hr. Immersion, max., per cent											
	Type I						Type II				Type III	
	Grade X	Grade P	Grade XX	Grade XXP	Grade XXX	Grade XXXXP	Grade C	Grade CE	Grade L	Grade LE	Grade A	Grade AA
1/8.....	8.0	7.0	3.1	3.1	1.8	1.8	...	...	6.0	4.0	3.0	...
1/16.....	6.0	5.0	2.0	2.0	1.2	1.2	4.4	1.8	2.5	1.8	1.5	1.0
3/16.....	4.2	3.6	1.6	1.6	1.0	1.0	3.2	1.5	1.9	1.5	1.1	1.5
1/4.....	3.3	2.8	1.3	1.3	0.85	0.85	2.5	1.4	1.6	1.25	0.95	0.95
5/16.....	2.3	2.1	1.0	1.0	0.70	0.70	1.9	1.2	1.3	1.0	0.85	0.80
3/8.....	1.8	1.7	0.85	0.85	0.60	0.60	1.6	1.0	1.1	0.90	0.70	0.70
1/2.....	1.1	...	0.55	...	0.45	...	1.2	0.70	0.80	0.65	0.55	0.55
5/8.....	0.85	...	0.50	...	0.40	...	1.1	0.65	0.75	0.60	0.50	0.50
1 and over....	0.75	...	0.45	...	0.35	...	1.0	0.60	0.70	0.55	0.45	0.45

<sup>a</sup> For intermediate thicknesses not given in this table, the value for the next smaller thickness applies.

TABLE VI.—DETAIL REQUIREMENTS FOR TUBES.

Type	Grade	Size, In- side by Outside Diameters, in.	Density <sup>a</sup> , min., g. per cu. cm.	Tensile Strength, min., psi.	Compressive Strength, Axial, min., psi.	Dielectric Strength Perpendicular to Lami- nations, min., v. per mil.		Water Absorption, max., per cent	Material As Received			After Immersion in Water for 24 hr. at 25 C.		
						Short-Time Test	Step-by-Step Test		Power Factor at 1,000,000 cycles, max.	Dielectric Constant at 1,000,000 cycles, max.	Loss Factor at 1,000,000 cycles, max.	Power Factor at 1,000,000 cycles, max.	Dielectric Constant at 1,000,000 cycles, max.	Loss Factor at 1,000,000 cycles, max.
I....	X rolled.....	1 by 1¼	1.10	7 500	10 000	500 <sup>b</sup>	300 <sup>b</sup>	5.0	0.040	5.0	0.20	0.070	6.0	0.42
	XX rolled.....	1 by 1¼	1.10	7 000	12 000	400	250	2.5	0.040	5.0	0.20	0.055	6.0	0.33
	X molded.....	1 by 1¼	1.25	9 000	15 000	400	250	4.0	0.045	6.0	0.26	0.070	7.5	0.52
	XX molded.....	1 by 1¼	1.25	7 500	15 000	300	200	2.0	0.040	5.5	0.22	0.055	6.5	0.35
II....	C rolled.....	1 by 1¼	1.10	5 500	11 000	none	none	3.0	...	...	...	...	...	...
	LE rolled.....	1 by 1¼	1.10	5 000	11 000	150	100	4.5	...	...	...	...	...	...
	LE rolled.....	1 by 1¼	1.10	5 000	11 000	150	100	2.5	...	...	...	...	...	...
	CE molded.....	1 by 1¼	1.25	6 500	19 000	175	100	1.5	...	...	...	...	...	...
	L molded.....	1 by 1¼	1.25	6 500	18 000	none	none	3.50	...	...	...	...	...	...
	L molded.....	1 by 1¼	1.25	6 500	18 000	none	none	1.75	...	...	...	...	...	...
	LE molded.....	1 by 1¼	1.25	6 000	19 000	150	90	2.20	...	...	...	...	...	...
	LE molded.....	1 by 1¼	1.25	6 000	19 000	175	110	1.10	...	...	...	...	...	...

<sup>a</sup> These density values apply to all sizes of tubes.

<sup>b</sup> The high dielectric strength of grade X rolled tubing is only obtainable under dry conditions. Under high humidity the dielectric strength of this grade is low.

standard length (Note 13), unless otherwise specified. When tubes cut to definite lengths are specified, the permissible variation shall be as shown in Table XI.

(b) *Diameter*.—The nominal inside and outside diameter (Note 14) shall be specified by the purchaser. The permissible variations in inside and outside diameters of round rolled and molded



TABLE VII.—DETAIL REQUIREMENTS FOR MOLDED RODS.

Type	Grade	Diameter, in.	Density, min., g. per cu.cm.	Tensile Strength, min., psi.	Flexural Strength, min., psi.	Compressive Strength (Ax- ial), min., psi.
I.....	XX.....	$\frac{1}{8}$ to 1, incl.....	1.30	8 500	15 000	20 000
		Over 1 to 2, incl.....	1.30	6 500	12 000	20 000
	XXX.....	$\frac{1}{8}$ to 1, incl.....	1.25	8 000	13 000	20 000
		Over 1 to 2, incl.....	1.25	6 000	11 000	20 000
II.....	C.....	$\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	1.28	7 500	17 000	20 000
		Over $\frac{1}{2}$ to 1, incl.....	1.28	8 000	17 000	20 000
		Over 1 to 2, incl.....	1.28	6 500	14 000	20 000
	CE.....	$\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	1.26	6 500	12 000	21 000
		Over $\frac{1}{2}$ to 1, incl.....	1.26	7 000	14 000	21 000
		Over 1 to 2, incl.....	1.26	6 000	12 000	21 000
	L.....	$\frac{3}{8}$ to 1, incl.....	1.28	8 000	16 000	20 000
		Over 1 to 2, incl.....	1.28	6 500	13 000	20 000
III.....	LE.....	$\frac{3}{8}$ to 1, incl.....	1.26	6 000	12 000	21 000
		Over 1 to 2, incl.....	1.26	4 800	9 000	21 000
	A.....	$\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	1.55	7 000	10 000	15 000
		Over $\frac{1}{2}$ to 1, incl.....	1.55	6 000	10 000	15 000
		Over 1 to 2, incl.....	1.55	5 000	9 000	15 000
	AA.....	$\frac{1}{2}$ to 1, incl.....	1.45	6 000	11 000	19 000
		Over 1 to 2, incl.....	1.45	5 500	9 000	19 000

TABLE VIII.—WATER ABSORPTION REQUIREMENTS FOR MOLDED RODS.

Diameter, in. <sup>a</sup>	Water Absorption, After 24-hr. Immersion, max., per cent							
	Type I		Type II				Type III	
	Grade XX	Grade XXX	Grade C	Grade CE	Grade L	Grade LE	Grade A	Grade AA
$\frac{1}{8}$ .....	.....	1.5	.....	1.5	1.5	1.2	2.0	.....
$\frac{1}{4}$ .....	1.5	1.0	2.5	1.5	1.2	0.90	1.5	1.25
$\frac{3}{8}$ .....	1.0	0.75	2.0	1.0	1.2	0.90	1.5	1.25
$\frac{1}{2}$ .....	1.0	0.75	2.0	1.0	1.2	0.90	1.5	1.25
Over 1 to 2, incl.....	1.3	0.75	1.5	1.2	1.2	1.1	1.75	1.75

<sup>a</sup> Intermediate diameters under 1 in. take the value for the next smaller diameter.

 TABLE IX.—PERMISSIBLE VARIATIONS IN SIZE OF  
PIECES CUT FROM STANDARD SHEETS.

Nominal Thickness, in.	Permissible Variations in Length or Width, Plus or Minus, in.		
	6 in. and under	6 to 24 in.	24 in. and over
0.010 to $\frac{1}{4}$ , incl.....	0.010	0.015	$\frac{1}{32}$
$\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	0.012	0.017	$\frac{1}{16}$
$\frac{1}{2}$ to 1, incl.....	0.015	0.020	$\frac{1}{8}$
1 to 1 $\frac{1}{2}$ , incl.....	0.018	0.030	$\frac{1}{4}$
1 $\frac{1}{2}$ to 2, incl.....	0.022	0.040	$\frac{1}{2}$

tubes shall be within the requirements prescribed in Table XII. The permissible variations in inside and outside dimensions of square and rectangular molded tubes shall be within the requirements prescribed in Table XIII.

(c) *Thickness.*—The permissible variations in wall thickness for round tubing

with inside diameters up to 4 in. shall be as shown in Table XIV. The permissible variations in wall thickness of square and rectangular molded tubes shall be within the requirements prescribed in Table XV.

NOTE 14.—Round tubes of inside diameters between 48 and 70 in. are available in a limited number of sizes. Standard steps of sizes of round tubes are as follows, except as limited by Table XVI:

Nominal Inside and Outside Diameters, in.	By Steps of, in.
$\frac{1}{8}$ to 1, incl.....	$\frac{1}{32}$
1 $\frac{1}{8}$ to 3, incl.....	$\frac{1}{16}$
3 $\frac{1}{8}$ to 6, incl.....	$\frac{1}{8}$
6 $\frac{1}{4}$ to 8, incl.....	$\frac{1}{4}$
8 to 25, incl.....	$\frac{1}{2}$

The standard sizes of square and rectangular molded tubes are as shown in Table XVII.



TABLE X.—PERMISSIBLE VARIATIONS IN THICKNESS OF SHEETS.\*

Nominal Thickness, in.		Permissible Variations, in.					
Fraction of Inch	Decimal Equivalent	Grades X, P, XX, XXX, XXXP, XXXP, A <sup>b</sup> Plus or Minus	Grade C Plus or Minus	Grade CE Plus or Minus	Grade L Plus or Minus	Grade LE Plus or Minus	Grade AA Plus or Minus
0.010	0.010	0.002	...	...	0.003	...	...
0.015	0.015	0.0025	...	...	0.0035	0.0035	...
0.020	0.020	0.003	...	...	0.004	0.004	...
0.025	0.025	0.0035	0.0065	0.0065	0.0045	0.0045	...
$\frac{1}{16}$	0.031	0.0035	0.0065	0.0065	0.005	0.005	...
$\frac{3}{64}$	0.047	0.0045	0.0075	...	0.0055	0.0055	...
$\frac{1}{8}$	0.0625	0.005	0.0075	0.0075	0.006	0.006	0.018
$\frac{5}{32}$	0.094	0.007	0.009	0.009	0.007	0.007	0.018
$\frac{3}{16}$	0.125	0.008	0.010	0.010	0.008	0.008	0.020
$\frac{1}{4}$	0.156	0.009	0.011	0.011	0.009	0.009	...
$\frac{5}{16}$	0.1875	0.010	0.0125	0.0125	0.010	0.010	0.024
$\frac{3}{8}$	0.219	0.011	0.014	0.014	0.011	0.011	...
		Plus or Minus	Plus Only	Plus or Minus	Plus Only	Plus or Minus	Plus or Minus
$\frac{1}{4}$	0.250	0.012	0.030	0.015	0.024	0.012	0.028
$\frac{3}{8}$	0.3125	0.0145	0.035	0.0175	0.029	0.0145	0.034
$\frac{1}{2}$	0.375	0.017	0.040	0.020	0.034	0.017	0.038
$\frac{3}{4}$	0.438	0.019	0.044	0.022	0.038	0.019	0.044
$\frac{1}{2}$	0.500	0.021	0.048	0.024	0.042	0.021	0.048
$\frac{3}{4}$	0.625	0.024	0.053	0.027	0.048	0.024	0.058
$\frac{1}{2}$	0.750	0.027	0.058	0.029	0.054	0.027	0.068
$\frac{3}{4}$	0.875	0.030	0.062	0.031	0.060	0.030	0.076
1	1.000	0.033	0.065	0.033	0.065	0.033	0.086
$1\frac{1}{16}$	1.125	0.035	0.069	0.035	0.069	0.035	...
$1\frac{1}{8}$	1.250	0.037	0.073	0.037	0.073	0.037	0.106
$1\frac{1}{4}$	1.375	0.039	0.077	0.039	0.077	0.039	...
$1\frac{1}{2}$	1.500	0.041	0.081	0.041	0.081	0.041	0.124
$1\frac{3}{4}$	1.625	0.043	0.085	0.043	0.085	0.043	...
$1\frac{7}{8}$	1.750	0.045	0.089	0.045	0.089	0.045	0.144
$1\frac{7}{8}$	1.875	0.047	0.093	0.047	0.093	0.047	...
2	2.000	0.049	0.097	0.049	0.097	0.049	0.160
$2\frac{1}{4}$	2.250	...	0.105	...	...	...	...
$2\frac{1}{2}$	2.500	...	0.113	...	...	...	...
$2\frac{3}{4}$	2.750	...	0.121	...	...	...	...
3	3.000	...	0.130	...	...	...	...
$3\frac{1}{2}$	3.500	...	0.146	...	...	...	...
4	4.000	...	0.163	...	...	...	...
$4\frac{1}{2}$	4.500	...	0.179	...	...	...	...
5	5.000	...	0.190	...	...	...	...
$5\frac{1}{2}$	5.500	...	0.210	...	...	...	...
6	6.000	...	0.230	...	...	...	...
$6\frac{1}{2}$	6.500	...	0.240	...	...	...	...
7	7.000	...	0.260	...	...	...	...
$7\frac{1}{2}$	7.500	...	0.280	...	...	...	...
8	8.000	...	0.290	...	...	...	...
$8\frac{1}{2}$	8.500	...	0.310	...	...	...	...
9	9.000	...	0.320	...	...	...	...
$9\frac{1}{2}$	9.500	...	0.340	...	...	...	...
10	10.000	...	0.360	...	...	...	...

\* On sheets of nominal thickness not listed in this table, the permissible variations shall be the same as for the next greater thickness.

<sup>b</sup> The minimum thickness for grades XXP, XXX, and XXXP is 0.015 in., and for grade A is 0.025 in. Grades P, XXP and XXXP are not available in thicknesses over  $\frac{1}{4}$  in.

### Rod Sizes and Permissible Variations in Dimensions

10. (a) *Length*.—Unless otherwise specified, molded rods shall be furnished to manufacturer's standard lengths (Note 15). When molded rods cut to definite lengths are specified, the permissible variations shall be as shown in Table XVIII.

NOTE 15.—Molded rods are available in lengths which vary from 18 to 48 in. in small diameters and from 30 to 48 in. in large diameters.

(b) *Diameter*.—The diameters of rods (Note 16) shall be as specified by the purchaser. The permissible variations in diameter of molded rods shall be as shown in Table XIX.

NOTE 16.—Molded rods are available in the following ranges of diameters:

Type	Grade	Range of Diameters, in.	
		Min.	Max.
I.....	XX, XXX.....	$\frac{1}{8}$	2
II.....	C, CE.....	$\frac{1}{4}$	4
	L, LE.....	$\frac{3}{8}$	4
III.....	A.....	$\frac{1}{4}$	2
	AA.....	$\frac{1}{2}$	2

### Sampling and Number of Tests

11. (a) *Sheets*.—A sheet shall be selected at random from a lot or shipment of material of a particular grade and thickness. One set of test specimens prescribed in the methods of testing

TABLE XI.—PERMISSIBLE VARIATIONS IN CUT LENGTHS OF TUBES.

Length, in.	Permissible Variations, Plus or Minus, in.		
	$\frac{3}{8}$ to 2 in., Outside Diameter	Over 2 to 4 in., Outside Diameter	Over 4 in., Outside Diameter
3 and under.....	0.010	0.010	0.030
Over 3 to 6, incl.....	0.010	0.015	0.030
Over 6 to 12, incl.....	0.015	0.020	0.030
Over 12 to 48, incl.....	0.030	0.030	0.050

(Section 13) shall be considered sufficient. The average result for the specimens tested shall conform to the requirements prescribed in these speci-

cations. Because of the expense in both material and time, it is recommended that complete conformance tests be confined where possible to sheets from  $\frac{1}{16}$  to  $\frac{1}{2}$  in. in thickness. Impact, flexural strength, and water absorption tests should serve well for routine control of uniformity of the mechanical grades. Water absorption and power factor tests will serve a similar purpose for the electrical grades.

(b) *Tubes*.—Random samples of any

TABLE XII.—PERMISSIBLE VARIATIONS IN DIAMETER OF ROUND TUBES.

Nominal Inside and Outside Diameters, in.	Permissible Variations, Plus or Minus, in.		
	Inside Diameter <sup>a</sup>		Outside Diameter
	Steel Mandrel	Built-up Mandrel	Ground, Buffed, or Varnished
$\frac{1}{8}$ to $\frac{23}{32}$ , incl.....	0.003	....	0.005
$\frac{3}{4}$ to $1\frac{1}{8}$ , incl.....	0.004	....	0.005
2 to 4, incl.....	0.008	....	0.008
Rolled Only: $4\frac{1}{2}$ to 12, incl.....	0.010	....	0.025
$12\frac{3}{4}$ to 18, incl.....	0.030	0.060	0.030 <sup>b</sup>
18 to 24, incl.....	0.040	0.075	0.035 <sup>b</sup>
24 to 48, incl.....	0.060	0.090	0.040 <sup>b</sup>

<sup>a</sup> While it is a commercial impracticability to maintain a complete range of mandrels in all sizes from 12 to 48 in. in diameter even in steps of 1 in., it is possible to make tubes on a built-up mandrel. In such cases this shall be accomplished by winding a phenolic laminated rolled tube on the steel mandrel, then curing and finishing the build-up to the required size. It is, of course, impossible to hold as close tolerances in inside diameter on tubes which are wound on a built-up mandrel as on a standard steel mandrel.

<sup>b</sup> Turned tubes.

TABLE XIII.—PERMISSIBLE VARIATIONS IN INSIDE AND OUTSIDE DIMENSIONS OF SQUARE AND RECTANGULAR MOLDED TUBES.<sup>a</sup>

Nominal Inside Dimension, in.	Permissible Variations in Inside Dimension, <sup>b</sup> Plus or Minus, in.	Nominal Outside Dimension, in.	Permissible Variations <sup>b</sup> in Outside Dimension, <sup>c</sup> Plus or Minus, in.	
	All Grades		Grades X, XX, XXX, A, L, and LE	Grades CE and AA
$\frac{1}{8}$ to $1\frac{1}{2}$ , incl.....	0.005	$\frac{3}{8}$ to $1\frac{1}{2}$ .....	0.010	0.017
$\frac{1}{2}$ to $3\frac{1}{2}$ , incl.....	0.006	$\frac{1}{2}$ to $3\frac{1}{2}$ .....	0.012	0.020
1 to $1\frac{1}{2}$ , incl.....	0.007	1 to $1\frac{1}{2}$ .....	0.014	0.022
2 to 4, incl.....	0.010	2 to 5.....	0.017	0.025

<sup>a</sup> These permissible variations apply to tubes of uniform nominal wall thickness in which the two axes perpendicular to the length are equal or which have a ratio one to the other not exceeding 4 to 1.

<sup>b</sup> Use variations corresponding with appropriate inside or outside dimension. For example, with a rectangular tube having nominal inside dimension  $\frac{1}{4}$  by  $1\frac{1}{2}$  in., the variation on  $\frac{1}{4}$  in. will be plus or minus 0.005 in. and on  $1\frac{1}{2}$  in. will be plus or minus 0.007 in.

<sup>c</sup> At the option of the manufacturer, outside dimension may be met by sanding or machining, if necessary.

grade and size of tubing may be taken to determine conformance with the density requirements. A minimum of 2 tubes from a lot of 50 tubes or fraction thereof, or 3 per cent of a larger quantity of tubes of any grade and size shall be tested for density. Other tests must be based on the sizes specified in Section 6. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. In the event the size of the test sample is not included in the order, the manufacturer may upon request provide proper size tubes prepared from the same materials

used in producing the tubing specified in the order. As an alternate, at the option of the purchaser, the manufacturer may submit detail limits for physical and electrical properties covering the size of tube in question.

(c) *Rods*.—Random samples of rods may be selected from a lot or shipment of any grade or size. A minimum of 2 rods from a lot of 50 rods or fraction thereof, or 3 per cent from larger lots shall be tested for density. For the other tests one set of test specimens as prescribed in the methods of testing (Section 15) shall be considered suffi-

TABLE XIV.—PERMISSIBLE VARIATIONS IN WALL THICKNESS<sup>a</sup> OF ROUND TUBES.

Nominal Wall Thickness, in.	Permissible Variations, Plus or Minus, in.							
	Rolled Tubes				Molded Tubes			
	Grade LE			Grades C, AA	Grades X, XX, XXX, L, LE, A			Grades CE, AA
Average for Single Tube	Grades X, XX, A	$\frac{3}{8}$ to $\frac{1}{2}$ in., Inside Diameter	Over $\frac{1}{2}$ in., Inside Diameter		$\frac{1}{8}$ to $\frac{1}{4}$ in., incl., Inside Diameter	Over $\frac{1}{4}$ to $\frac{1}{2}$ in., incl., Inside Diameter	Over $\frac{1}{2}$ in., Inside Diameter	
Under $\frac{1}{8}$ .....	0.006	0.010	0.008	not made	0.008	0.008	0.008	
$\frac{1}{8}$ to $\frac{3}{8}$ , excl.....	0.007	0.011	0.009	0.015	0.011	0.011	0.011	0.015
$\frac{3}{8}$ to $\frac{1}{2}$ , excl.....	0.009	0.013	0.011	0.020	0.017	0.015	0.011	0.020
$\frac{1}{2}$ to $\frac{3}{4}$ , incl.....	0.011	0.015	0.013	0.020	0.025	0.018	0.013	0.020

<sup>a</sup> Wall thickness measured at any point in any wall of any one tube of a given size shall fall within these permissible variations. This provides a means for measuring both the variation in wall thickness and deviation from parallelism.

TABLE XV.—PERMISSIBLE VARIATIONS IN WALL THICKNESS<sup>a</sup> OF SQUARE AND RECTANGULAR MOLDED TUBES.

Wall Thickness, in.	Permissible Variation in Wall Thickness, <sup>b</sup> Plus or Minus, in.				
	Grades X, XX, XXX, A, L, and LE			Grade CE	Grade AA
	Inside Dimension <sup>c</sup>			Inside Dimension <sup>c</sup>	
Average for Single Tube	$\frac{3}{8}$ to $\frac{1}{4}$ in.	$\frac{1}{4}$ to $\frac{1}{2}$ in., incl.	Over $\frac{1}{2}$ in.	Over $\frac{3}{8}$ in.	Over $\frac{1}{2}$ in.
$\frac{3}{8}$ to $\frac{1}{4}$ , excl.....	0.008	0.008	0.010	.....	.....
$\frac{1}{4}$ to $\frac{3}{8}$ , excl.....	0.011	0.010	0.013	0.015	0.025
$\frac{3}{8}$ to $\frac{1}{2}$ , excl.....	0.015	0.014	0.016	0.020	0.030
$\frac{1}{2}$ to $\frac{3}{4}$ , incl.....	.....	0.018	0.020	0.025	0.030

<sup>a</sup> Wall thickness measured at any point in any wall of any one tube of a given size shall fall within these permissible variations. This provides a means for measuring both the variation in wall thickness and deviation from parallelism.

<sup>b</sup> Tubes with a wall thickness greater than one half of the minimum inside dimension cannot be expected to meet these permissible variations. At the option of the manufacturer, outside dimension may be met by sanding or machining, if necessary.

<sup>c</sup> Use variations corresponding with maximum inside dimension.

cient. The average result for the specimens tested shall conform to the requirements prescribed in these specifications.

### Conditioning

12. (a) *Conditioning Test Specimens*.—When conditioning is specified the material shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials,<sup>3</sup> except as modified in the testing methods or in these specifications.

(b) *Test Conditions*.—Tests shall be conducted in a laboratory atmosphere of  $25 \pm 8$  C. ( $77 \pm 15$  F.), unless otherwise

specified in the testing methods or in these specifications.

### Methods of Testing Sheets

13. The properties for sheets enume-

edge to measure warp. The twist shall be determined by suspending the sheet vertically from adjacent corners in succession and measuring the deviation along the diagonal from a horizontal

TABLE XVI.—AVAILABLE SIZES OF TUBES (SEE NOTE 14).

Type	Grade	Rolled Tubes						Molded Tubes					
		Inside Diameter, in.		Outside Diameter, in.		Wall Thickness, in.		Inside Diameter, in.		Outside Diameter, in.		Wall Thickness, in.	
		Min.	Max.	Min.	Max.	Min.	Max. <sup>a</sup>	Min.	Max.	Min.	Max.	Min.	Max. <sup>a</sup>
I.....	X.....	1/8	48	3/16	50	1/16	3/4	1/4	37/8	1/4	4	1/16	3/4
	XX.....	1/8	48	3/16	50	1/16	1	1/4	37/8	1/4	4	1/16	1
	XXX.....	none		none		..		1/8	37/8	1/4	4	1/16	1
II.....	C.....	3/8	48	1/2	50	1/16	2	none		none		1/16	1 1/2
	CE.....	none		none		..		1/8	37/8	3/8	4	1/16	1 1/2
	L.....	none		none		..		1/8	37/8	3/8	4	1/16	1
III.....	LE.....	3/8	48	1/4	50	1/16	1	1/8	37/8	3/8	4	1/16	1
	A.....	3/16	48	3/8	50	1/16	1 1/2	3/8	37/8	1/2	4	1/16	1 1/2
	AA.....	1	48	1 1/4	50	3/8	2	3/8	37/8	1/2	4	3/8	1 1/2

<sup>a</sup> Tubes with a wall thickness greater than one half of the minimum inside dimension cannot be recommended for many applications.

TABLE XVII.—STANDARD SIZES OF SQUARE AND RECTANGULAR MOLDED TUBES.

Type	Grade	Inside Dimension, in.		Outside Dimension, in.		Wall Thickness, in.	
		Min.	Max.	Min.	Max.	Min.	Max. <sup>a</sup>
I.....	X.....	3/16	3 3/4	9/16	4	3/64	1/2
	XX.....	3/16	3 3/4	9/16	4	3/64	1 1/2
	XXX.....	3/16	3 3/4	9/16	4	3/64	1 1/2
II.....	CE.....	3/8	3 3/4	1 1/2	4	1/16	1 1/2
	L.....	3/8	3 3/4	1 1/2	4	1/16	1 1/2
	LE.....	3/8	3	1 1/2	4	3/64	1 1/2
III.....	A.....	1/4	3 3/4	1 1/2	4	1/16	1 1/2
	AA.....	1/2	3 3/4	3/4	4	3/8	1 1/2

<sup>a</sup> Tubes with a wall thickness greater than one half of the minimum inside dimension cannot be recommended for many applications.

TABLE XVIII.—PERMISSIBLE VARIATIONS IN CUT LENGTHS OF MOLDED RODS.

Length, in.	Permissible Variations, Plus or Minus, in.	
	1/4 to 1 1/4 in., incl., in Diameter	2 to 4 in., incl., in Diameter
3 and under.....	0.010	0.010
Over 3 to 6, incl.....	0.010	0.015
Over 6 to 12, incl.....	0.015	0.020
Over 12.....	0.030	0.030

TABLE XIX.—PERMISSIBLE VARIATIONS IN DIAMETER OF MOLDED RODS.

Nominal Diameter, in.	Permissible Variation, Plus or Minus, in.
1/8 to 1 15/16, incl.....	0.005
2 to 4, incl.....	0.008

rated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Warp or Twist*.—The warp or twist shall be determined on the sheet in the as-received condition by laying a straight edge along the dimension to be measured, and measuring the greatest deviation by use of a metal scale. Sheets shall be suspended in a vertical position against a horizontal straight

edge. Calculate the warp or twist as follows:

$$W = \frac{D \times 100}{L}$$

where:

$W$  = percentage warp or twist,  
 $D$  = maximum deviation of sheet from straight edge in inches, and  
 $L$  = length of sheet in direction measured in inches.

In order to compare the relative warp or twist for any length of sheet, calculate the percentage warp or twist, respectively, for a 36-in. length as follows:

$$C = \frac{W \times 36}{L}$$

where:

$C$  = percentage warp or twist calculated for a 36-in. length,

$W$  = percentage warp or twist, and

$L$  = length of sheet in direction measured in inches.

(b) *Tensile and Compressive Strengths*.—Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229).<sup>3</sup> Specimens shall be tested in the as-received condition.

(c) *Flexural Strength*.—Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229).<sup>3</sup> Specimens shall be tested in the as-received condition.

(d) *Impact Strength*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup> except that the time of conditioning shall be 48 hr. for any thickness of test specimen, and the specimens shall be cooled in a desiccator to room temperature before testing.

(e) *Power Factor, Dielectric Constant, and Loss Factor*.—Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup> Specimens shall be tested in the as-received condition.

(f) *Dielectric Strength*.—Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229).<sup>3</sup> The dielectric strength perpendicular to laminations only shall be determined,

and specimens shall be tested in the as-received condition.

(g) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup> The test specimens shall be conditioned for 1 hr. at 105 to 110 C. (221 to 230 F.) prior to the 24-hr. immersion.

(h) *Dimensions (Length, Width, and Thickness)*.—The length and width of sheet shall be determined on the material in the as-received condition by means of a steel scale. The thickness shall be determined in accordance with the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229).<sup>3</sup>

#### Methods of Testing Tubes

14. The properties of tubes enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Warp or Twist*.—Standard Methods of Measuring Dimensions of Rigid Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 668).<sup>3</sup> except that the warp shall be measured on tubes in the as-received condition.

(b) *Tensile and Compressive Strength*.—Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 348).<sup>3</sup>

(c) *Power Factor, Dielectric Constant, and Loss Factor*.—Section 12 (b) of the Tentative Specifications for Round Phenolic Laminated Tubing for Radio Applications (A.S.T.M. Designation: D 616).<sup>3</sup>

(d) *Dielectric Strength*.—Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 348).<sup>3</sup> except that the specimens shall be tested in the as-received condition.

(e) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup> The test specimens shall be conditioned for 1 hr. at 105 to 110 C. (221 to 230 F.) prior to the 24-hr. immersion.

(f) *Dimensions*.—Standard Methods of Measuring Dimensions of Rigid Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 668).<sup>3</sup>

#### Methods of Testing Rods

15. The properties for rods enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Warp or Twist*.—Tentative Methods of Measuring Dimensions of Rigid Rods Used in Electrical Insulation (A.S.T.M. Designation: D 741),<sup>3</sup> except that the warp shall be measured on rods in the as-received condition.

(b) *Tensile and Compressive Strengths*.—Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (A.S.T.M. Designation: D 349).<sup>3</sup>

(c) *Flexural Strength*.—Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (A.S.T.M. Designation: D 349).<sup>3</sup>

(d) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup> The test specimens shall be conditioned for 1 hr. at 105 to 110 C. (221 to 230 F.) prior to the 24-hr. immersion.

(e) *Dimensions*.—Tentative Methods of Measuring Dimensions of Rigid Rods

Used in Electrical Insulation (A.S.T.M. Designation: D 741).<sup>3</sup>

#### Retest and Rejection

16. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser. Any portion of an accepted shipment of material which is subsequently found not to be in accordance with these specifications may be rejected provided the manufacturer is notified within 90 days from the date of receipt of the material by the purchaser.

#### Packing and Marking

17. (a) *Packing*.—The material shall be packed in substantial crates, boxes, or cartons so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, form, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.



## APPENDIX

## DETAILED DESCRIPTIONS OF THE VARIOUS GRADES OF LAMINATED MATERIALS

*Type I.—Cellulose paper base laminated material:*

**Grade X.**—A paper base laminated material primarily intended for mechanical applications where electrical requirements are of secondary importance. Should be used with discretion when high humidity conditions are encountered. Not equal to fabric base grades in impact strength.

**Grade P.**—A paper base laminated material primarily intended for punching. More flexible and not quite as strong as grade X. Moisture resistance and electrical properties intermediate between grades X and XX.

**Grade XX.**—A paper base laminated material suitable for usual electrical applications. Good machinability.

**Grade XXP.**—A paper base laminated material similar to grade XX in electrical and moisture resisting properties, but more suitable for hot punching. Intermediate between grades P and XX in punching and cold flow characteristics.

**Grade XXX.**—A paper base laminated material better electrically than grade XX and suitable for high humidity applications, and with minimum cold flow characteristics.

**Grade XXXP.**—A paper base laminated material similar to grade XXX, but having lower dielectric losses and being more suitable for hot punching. This grade has greater cold flow than grade XXX, and is intermediate between grades XXP and XXX in punching characteristics.

*Type II.—Cellulose fabric base laminated material:*

**Grade C.**—A fabric base laminated material made throughout from cotton fabric weighing over 4 oz. per sq. yd. and having a count as determined from inspection of the laminated plate of not more than 72 threads per inch in the filler direction, nor more than 140 threads per inch total in both warp and filler directions. A strong, tough material suitable for gears and other applications requiring high impact. The heavier the fabric base used the higher will be the impact strength, but the rougher the

machined edge; consequently, there may be several subclasses in this grade adapted for various sizes of gears and types of mechanical service. Should not be used for electrical applications except for low voltages.

**Grade CE.**—A fabric base laminated material of the same fabric weight, and thread count as grade C. For electrical applications requiring greater toughness than grade XX, or mechanical applications requiring greater resistance to moisture than grade C. Exceptionally good in moisture resistance.

**Grade L.**—A fine weave fabric base laminated material made throughout from cotton fabric weighing 4 oz. or less per square yard. As determined by inspection of the laminated plate, the minimum thread count per inch in any ply shall be 72 in the filler direction and 140 total in both warp and filler directions. For purpose of identification, the surface sheets shall have a minimum thread count of 80 threads per inch in each of the warp and filler directions. This grade is suitable for small gears and other fine machining applications, particularly in thickness under  $\frac{1}{8}$  in. Not quite as tough as grade C. Should not be used for electrical application except for low voltage.

**Grade LE.**—A fine weave fabric base laminated material of the same fabric weight, and thread count as grade L. For electrical applications requiring greater toughness than grade XX. Better machining properties and finer appearance than grade CE, also available in thinner sizes. Exceptionally good in moisture resistance.

*Type III.—Asbestos base laminated material:*

**Grade A.**—An asbestos paper base laminated material. More resistant to flame and slightly more resistant to heat than other laminated grades because of high inorganic content. Suitable for only low voltage applications. Minimum dimensional changes when exposed to moisture.

**Grade AA.**—An asbestos fabric base laminated material. Similar to grade A but stronger and tougher. Minimum dimensional changes when exposed to moisture.

## *Tentative Specifications for*

### VULCANIZED FIBRE SHEETS, RODS, AND TUBES USED FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 710 - 43 T

ISSUED, 1943.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. These specifications cover vulcanized fibre sheets, round rods, and round tubes of such grades suitable for use as electrical insulation.

#### Vulcanized Fibre

2. Vulcanized fibre is made by combining layers of chemically gelled paper. The chemical compound used in gelling the paper is subsequently removed by leaching, and the resulting product after being dried and finished by calendering, is a dense material of partially regenerated cellulose in which the fibrous structure is retained.

#### Grades

3. Three grades of vulcanized fibre are covered, as follows:

*Bone Grade.*—Bone grade vulcanized fibre is characterized by high

density resulting from modification of the cellulose. It possesses maximum hardness and physical strength as well as excellent turning, threading, and milling characteristics.

*Commercial Grade.*—Commercial grade vulcanized fibre is generally characterized by lower density than bone grade. It possesses good physical and electrical properties and can be fabricated satisfactorily by punching, turning, and forming operations. It is intended for general mechanical and electrical applications and is sometimes referred to as Mechanical and Electrical Grade.

*Electrical Insulation Grade.*—Electrical insulation grade vulcanized fibre is characterized by high dielectric strength and toughness. It is primarily intended for specific electrical applications, particularly those involving difficult forming or bending operations, and it is sometimes referred to as "fish paper."

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, April 27, 1943.



**Forms and Colors**

4. Vulcanized fibre in the following forms and colors are covered:

	Forms	Colors <sup>a</sup>
Bone Grade.....	Sheets, <sup>b</sup> tubes, rods	Gray
Commercial Grade.....	Sheets, <sup>b</sup> tubes, rods	Red, Gray, Black
Electrical Insulation Grade.....	Sheets <sup>b</sup>	Gray

<sup>a</sup> In any of these standard colors, considerable variation in shade may be expected.

<sup>b</sup> Sheets are available both flat and in rolls. Rolls are made of sheets in certain thin thicknesses only, generally one continuous sheet per roll.

**Chemical Requirements**

5. The material shall conform to the requirements as to chemical composition prescribed in Table I.

TABLE I.—CHEMICAL REQUIREMENTS.

Grade	Color	Zinc Chloride, max., per cent	Ash, max., per cent
Bone.....	Gray.....	0.2	3
Commercial.....	Red.....	0.2	7
	Gray and Black...	0.2	3
Electrical Insulation...	Gray.....	0.2	3

**Detail Requirements for Round Tubes**

8. Round tubes shall conform to the requirements as to physical and electrical properties prescribed in Tables IX

TABLE III.—WATER ABSORPTION AND DIELECTRIC STRENGTH REQUIREMENTS FOR SHEETS.

Grade	Nominal Thickness, in.	Water Absorption, max., per cent,		Dielectric Strength, min., v. per mil.
		2 hr.	24 hr.	
Bone.....	$\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	..	..	175
	$\frac{1}{4}$ to $\frac{3}{8}$ , incl.....	50	60	..
	Over $\frac{3}{8}$ to $\frac{1}{2}$ , incl.....	..	..	150
	Over $\frac{1}{2}$ to $\frac{3}{4}$ , incl.....	20	50	100
	Over $\frac{3}{4}$ to $1$ , incl.....	10	25	50
Commercial.....	Up to $\frac{1}{8}$ , incl.....	..	..	175
	$\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	55	65	..
	Over $\frac{1}{4}$ to $\frac{3}{8}$ , incl.....	..	..	150
	Over $\frac{3}{8}$ to $\frac{1}{2}$ , incl.....	25	60	100
	Over $\frac{1}{2}$ to $1$ , incl.....	15	35	50
	Over $1$ to $2$ , incl.....	15	35	..
	Over $2$ to $3$ , incl.....	8	20	a
Electrical Insulation.	0.004 to 0.005, incl.....	..	..	200
	Over 0.005 to 0.015, incl.....	..	..	300
	Over 0.015 to 0.040, incl.....	..	..	250
	Over 0.040 to $\frac{1}{8}$ , incl.....	..	..	175
	$\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	55	65	..

<sup>a</sup> Total breakdown voltage of 25,000 v.

TABLE II.—TENSILE, COMPRESSIVE, AND FLEXURAL STRENGTH REQUIREMENTS FOR SHEETS.

Grade	Nominal Thickness, in.	Tensile Strength, min., psi.		Compressive Strength, min., psi.	Flexural Strength, min., psi.	
		Crosswise	Lengthwise	Flatwise	Crosswise	Lengthwise
Bone.....	$\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	6 500	8 500	30 000	..	..
	Over $\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	6 000	8 000	30 000	13 000	15 000
Commercial.....	Up to $\frac{1}{8}$ , incl.....	6 000	8 000	20 000	..	..
	Over $\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	5 500	7 500	20 000	12 000	14 000
	Over $\frac{1}{4}$ to $1$ , incl.....	5 000	7 000	20 000	11 000	13 000
Electrical Insulation...	Up to $\frac{1}{8}$ , incl.....	6 000	8 000	..	..	..

**Detail Requirements for Sheets**

6. Sheet material shall conform to the requirements as to physical and electrical properties prescribed in Tables II, III, IV, and V.

**Detail Requirements for Round Rods**

7. Round rods shall conform to the requirements as to physical properties prescribed in Tables VI, VII, and VIII.

and X. Density of tubing shall conform to the requirements prescribed in Table V for the respective grade and thickness.

**Sheet Sizes and Permissible Variations**

9. (a) Sheets shall be furnished in the manufacturer's standard sheet sizes.

NOTE.—In general, manufacturer's standard sizes for the various grades and thicknesses will be as shown in Table XI.

(b) When sheets and rolls are trimmed to a specified width, the maximum permissible variation in width shall be plus or minus  $\frac{1}{2}$  in.

TABLE IV.—BURSTING AND TEARING STRENGTH REQUIREMENTS FOR ELECTRICAL INSULATION GRADE SHEETS.

Grade	Nominal Thickness, in.	Bursting Strength, min., psi.		Tearing Strength, min., g.	
			Machine Direction	Cross Direction	
Electrical Insulation....	0.005....	75	100	120	
	0.007....	105	190	220	
	0.010....	150	250	300	
	0.015....	225	375	450	
	0.020....	325	...	...	

TABLE V.—DENSITY REQUIREMENTS FOR SHEETS.

Grade	Nominal Thickness, in.	Density, min., g. per cu. cm.
Bone.....	$\frac{1}{8}$ to $\frac{1}{2}$ incl.....	1.30
Commercial....	Up to 0.010.....	0.90
	0.010 to $\frac{1}{8}$ , incl.....	1.15
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	1.20
	Over $\frac{1}{2}$ to 1, incl.....	1.10
	Over 1 to $1\frac{1}{4}$ incl.....	1.05
Electrical Insulation.....	Over $1\frac{1}{4}$ .....	1.01
	Up to 0.010.....	0.90
	0.010 to $\frac{1}{8}$ , incl.....	1.15
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	1.20

TABLE VI.—TENSILE, COMPRESSIVE, AND FLEXURAL STRENGTH REQUIREMENTS FOR ROUND RODS.

Grade	Nominal Diameter, in.	Tensile Strength, min., psi.	Compressive Strength, min., psi.	Flexural Strength, min., psi.
Bone.....	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	8 000	8 000	15 000
Commercial....	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	7 500	6 000	14 000
	Over $\frac{1}{2}$ to 1, incl.....	7 000	6 000	13 000

(c) When sheets are trimmed to a specified length, the maximum permissible variation in length shall be plus or minus  $\frac{1}{2}$  in.

(d) The maximum permissible variations in widths of strips cut from sheets

TABLE VII.—WATER ABSORPTION REQUIREMENTS FOR ROUND RODS.

Grade	Nominal Diameter, in.	Water Absorption, max., per cent	
		2 hr.	24 hr.
Bone.....	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	35	75
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	15	50
Commercial....	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	40	80
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	20	60
	Over $\frac{1}{2}$ to 1, incl.....	10	30
	Over 1 to 2, incl.....	8	25

TABLE VIII.—DENSITY REQUIREMENTS FOR ROUND RODS.

Grade	Nominal Diameter, in.	Density, min., g. per cu. cm.
Bone.....	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	1.30
Commercial....	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	1.15
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	1.20
	Over $\frac{1}{2}$ to 1, incl.....	1.10
	Over 1 to $1\frac{1}{4}$ , incl.....	1.05
	Over $1\frac{1}{4}$ to 2, incl.....	1.01

TABLE IX.—TENSILE AND COMPRESSIVE STRENGTH REQUIREMENTS FOR ROUND TUBES.

Grade	Nominal Wall Thickness, in.	Tensile Strength, min., psi.	Axial Compressive Strength, min., psi.
Bone and Commercial	All sizes.....	6500	...
	Up to 1.5 in. in outside diameter	...	10 000
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	...	9 000

TABLE X.—WATER ABSORPTION AND DIELECTRIC STRENGTH REQUIREMENTS FOR ROUND TUBES.

Grade	Nominal Wall Thickness, in.	Water Absorption, max., per cent		Dielectric Strength, min., v. per mil.
		2 hr.	24 hr.	
Bone and Commercial	Up to $\frac{1}{8}$ .....	..	..	175
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	..	..	150
	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	50	75	...
	Over $\frac{1}{2}$ to $1\frac{1}{4}$ , incl.....	20	50	100
	Over $\frac{1}{4}$ to $\frac{3}{8}$ , incl.....	10	25	50

by the indicated operations shall be as prescribed in Table XII.

(e) The maximum permissible vari-

ation in thickness of full sheets shall be as prescribed in Table XIII.

TABLE XI.—MANUFACTURER'S STANDARD SIZES OF SHEETS.<sup>a</sup>

Grade	Thickness, in.	Width, in.	Length, in.
Bone.....	$\frac{1}{32}$ to $\frac{1}{16}$ , incl.....	40 to 46	80 to 84
	Over $\frac{1}{16}$ to $\frac{1}{8}$ , incl.....	40 to 46	66 to 84
Commercial...{	0.010 to $\frac{1}{16}$ incl.....	45 to 52	80 to 84
	Over $\frac{1}{16}$ to 2, incl.....	45 to 52	72 to 84
Electrical Insulation }...	0.004 to $\frac{1}{8}$ , incl.....	46 to 52	80 to 84

<sup>a</sup> Other standard sizes of sheets can be furnished by some manufacturers ranging up to 50 in. in width and 90 in. in length in the bone grade, 56 in. in width by 90 in. in length in the commercial grade, and 56 in. in width in the electrical insulation grade.

TABLE XII.—PERMISSIBLE VARIATIONS IN WIDTHS OF STRIP CUT FROM SHEETS.<sup>a</sup>

Width, in.	Permissible Variations, plus or minus, in.			
	Slit (Ribbon Rolls)	Sheared	Band Sawed	Smooth Sawed
$\frac{1}{32}$ and under.....	0.010	0.015	0.020	0.006
$\frac{1}{16}$ to $\frac{1}{8}$ , incl.....	0.015	0.020	0.030	0.008
$\frac{1}{8}$ to 1, incl.....	0.020	0.030	0.045	0.010
$\frac{1}{4}$ to 2, incl.....	0.030	0.040	0.060	0.012
Over 2 to 4.....	0.040	0.050	0.075	0.014

<sup>a</sup> On strip of widths not listed in this table, the permissible variations shall be the same as for the next greater width.

TABLE XIII.—PERMISSIBLE VARIATIONS IN THICKNESS OF FULL SHEETS.<sup>a</sup>

Nominal Thickness, in.	Permissible Variations, plus or minus, in.
0.004 to 0.040, incl.....	10 per cent
$\frac{1}{32}$ .....	0.004
0.050 to $\frac{1}{16}$ , incl.....	0.005
0.065 to $\frac{1}{8}$ , incl.....	0.007
0.100 to $\frac{1}{4}$ , incl.....	0.009
$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	0.012
$\frac{1}{4}$ to $\frac{3}{4}$ , incl.....	0.015
$\frac{1}{2}$ to 1, incl.....	0.025
$\frac{3}{4}$ to 1 $\frac{1}{2}$ , incl.....	0.040
1 $\frac{1}{2}$ to 2, incl.....	0.060

<sup>a</sup> On sheets of nominal thickness not listed in this table, the permissible variations shall be the same as for the next greater thickness.

(f) The maximum permissible variations in sheets cut in halves, thirds, or quarters shall be as shown in Table XIV.

#### Rod Sizes and Permissible Variations

10. (a) Rods shall be furnished in the same nominal sizes as sheets. Rods

shall be cut from sheet and the length is limited by the length of the sheet.

(b) The maximum permissible variations in diameters of rods shall be as shown in Table XV.

TABLE XIV.—PERMISSIBLE VARIATIONS IN THICKNESS OF SHEETS CUT IN HALVES, THIRDS, AND QUARTERS.

Nominal Thickness, in.	Permissible Variations, plus or minus, in.
$\frac{1}{32}$ and under.....	8 per cent
Over $\frac{1}{32}$ to $\frac{1}{16}$ , incl.....	0.004
Over $\frac{1}{16}$ to $\frac{1}{8}$ , incl.....	0.005
Over $\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	0.007
Over $\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	0.010
Over $\frac{1}{2}$ to 1, incl.....	0.012
Over 1 to 2, incl.....	0.012
Over 2 to 4, incl.....	0.018
Over 4 to 8, incl.....	0.020
Over 8 to 16, incl.....	0.030
Over 16.....	0.040

TABLE XV.—PERMISSIBLE VARIATIONS IN DIAMETER OF RODS.<sup>a</sup>

Nominal Size of Rod, Outside Diameter, in.	Permissible Variations, plus or minus, in.
$\frac{1}{32}$ and under.....	0.005
$\frac{1}{16}$ to 1, incl.....	0.006
$\frac{1}{8}$ to 2, incl.....	0.010

<sup>a</sup> On rods of nominal diameters not listed in this table, the permissible variations shall be the same as for the next greater diameter.

TABLE XVI.—STANDARD STEPS IN INSIDE AND OUTSIDE DIAMETERS OF TUBES.

Nominal Inside Diameter, in.	Diameters Available in Steps of, in.	Nominal Outside Diameter, in.	Diameters Available in Steps of, in.
$\frac{1}{8}$ to $\frac{3}{8}$ , incl.....	$\frac{1}{64}$	$\frac{3}{8}$ to $\frac{1}{2}$ , incl.....	$\frac{1}{64}$
$\frac{1}{2}$ to 1, incl.....	$\frac{1}{32}$	$\frac{1}{2}$ to 1 $\frac{1}{4}$ , incl.....	$\frac{1}{32}$
$\frac{1}{16}$ and over.....	$\frac{1}{16}$	$\frac{1}{16}$ and over.....	$\frac{1}{16}$

#### Tube Sizes and Permissible Variations

11. (a) The sizes of tubing shall be as shown in Tables XVI, and XVII.

(b) The maximum permissible variations in inside and outside diameters of tubes shall be as shown in Table XVIII.

#### Methods of Testing

12. Vulcanized fibre shall be conditioned and tested in accordance with

the Tentative Methods of Testing Vulcanized Fibre Used for Electrical Insulation (A.S.T.M. Designation: D 619).<sup>3</sup>

### Workmanship

13. The material shall be uniform in quality, consistent with the properties prescribed in these specifications. It

TABLE XVII.—WALL THICKNESS OF TUBES.<sup>a</sup>

Nominal Inside Diameter, in.	Wall Thickness, in.	
	Minimum	Maximum
1/8	0.020	1/8
3/16	0.020	3/16
1/4	0.020	1/4
5/16	0.020	5/16
3/8	0.020	3/8
1/2	0.020	1/2
5/8	1/32	5/8
3/4	1/16	3/4
7/8	1/16	7/8
1	1/16	1
1 1/8	1/16	1 1/8
1 1/4	1/16	1 1/4
1 1/2	1/16	1 1/2
1 3/4	1/16	1 3/4
2	1/16	2
2 1/4	1/16	2 1/4
2 1/2	1/16	2 1/2
2 3/4	1/16	2 3/4
3	1/16	3
3 1/4	1/16	3 1/4
3 1/2	1/16	3 1/2
3 3/4	1/16	3 3/4
4	1/16	4
4 1/4	1/16	4 1/4
4 1/2	1/16	4 1/2
4 3/4	1/16	4 3/4
5	1/16	5
5 1/4	1/16	5 1/4
5 1/2	1/16	5 1/2
5 3/4	1/16	5 3/4
6	1/16	6
6 1/4	1/16	6 1/4
6 1/2	1/16	6 1/2
6 3/4	1/16	6 3/4
7	1/16	7
7 1/4	1/16	7 1/4
7 1/2	1/16	7 1/2
7 3/4	1/16	7 3/4
8	1/16	8
8 1/4	1/16	8 1/4
8 1/2	1/16	8 1/2
8 3/4	1/16	8 3/4
9	1/16	9
9 1/4	1/16	9 1/4
9 1/2	1/16	9 1/2
9 3/4	1/16	9 3/4
10	1/16	10
10 1/4	1/16	10 1/4
10 1/2	1/16	10 1/2
10 3/4	1/16	10 3/4
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55 1/4	1/16	55 1/4
55 1/2	1/16	55 1/2
55 3/4	1/16	55 3/4
56	1/16	56
56 1/4	1/16	56 1/4
56 1/2	1/16	56 1/2
56 3/4	1/16	56 3/4
57	1/16	57
57 1/4	1/16	57 1/4
57 1/2	1/16	57 1/2
57 3/4	1/16	57 3/4
58	1/16	58
58 1/4	1/16	58 1/4
58 1/2	1/16	58 1/2
58 3/4	1/16	58 3/4
59	1/16	59
59 1/4	1/16	59 1/4
59 1/2	1/16	59 1/2
59 3/4	1/16	59 3/4
60	1/16	60
60 1/4	1/16	60 1/4
60 1/2	1/16	60 1/2
60 3/4	1/16	60 3/4
61	1/16	61
61 1/4	1/16	61 1/4
61 1/2	1/16	61 1/2
61 3/4	1/16	61 3/4
62	1/16	62
62 1/4	1/16	62 1/4
62 1/2	1/16	62 1/2
62 3/4	1/16	62 3/4
63	1/16	63
63 1/4	1/16	63 1/4
63 1/2	1/16	63 1/2
63 3/4	1/16	63 3/4
64	1/16	64
64 1/4	1/16	64 1/4
64 1/2	1/16	64 1/2
64 3/4	1/16	64 3/4
65	1/16	65
65 1/4	1/16	65 1/4
65 1/2	1/16	65 1/2
65 3/4	1/16	65 3/4
66	1/16	66
66 1/4	1/16	66 1/4
66 1/2	1/16	66 1/2
66 3/4	1/16	66 3/4
67	1/16	67
67 1/4	1/16	67 1/4
67 1/2	1/16	67 1/2
67 3/4	1/16	67 3/4
68	1/16	68
68 1/4	1/16	68 1/4
68 1/2	1/16	68 1/2
68 3/4	1/16	68 3/4
69	1/16	69
69 1/4	1/16	69 1/4
69 1/2	1/16	69 1/2
69 3/4	1/16	69 3/4
70	1/16	70
70 1/4	1/16	70 1/4
70 1/2	1/16	70 1/2
70 3/4	1/16	70 3/4
71	1/16	71
71 1/4	1/16	71 1/4
71 1/2	1/16	71 1/2
71 3/4	1/16	71 3/4
72	1/16	72
72 1/4	1/16	72 1/4
72 1/2	1/16	72 1/2
72 3/4	1/16	72 3/4
73	1/16	73
73 1/4	1/16	73 1/4
73 1/2	1/16	73 1/2
73 3/4	1/16	73 3/4
74	1/16	74
74 1/4	1/16	74 1/4
74 1/2	1/16	74 1/2
74 3/4	1/16	74 3/4
75	1/16	75
75 1/4	1/16	75 1/4
75 1/2	1/16	75 1/2
75 3/4	1/16	75 3/4
76	1/16	76
76 1/4	1/16	76 1/4
76 1/2	1/16	76 1/2
76 3/4	1/16	76 3/4
77	1/16	77
77 1/4	1/16	77 1/4
77 1/2	1/16	77 1/2
77 3/4	1/16	77 3/4
78	1/16	78
78 1/4	1/16	78 1/4
78 1/2	1/16	78 1/2
78 3/4	1/16	78 3/4
79	1/16	79
79 1/4	1/16	79 1/4
79 1/2	1/16	79 1/2
79 3/4	1/16	79 3/4
80	1/16	80
80 1/4	1/16	80 1/4
80 1/2	1/16	80 1/2
80 3/4	1/16	80 3/4
81	1/16	81
81 1/4	1/16	81 1/4
81 1/2	1/16	81 1/2
81 3/4	1/16	81 3/4
82	1/16	82
82 1/4	1/16	82 1/4
82 1/2	1/16	82 1/2
82 3/4	1/16	82 3/4
83	1/16	83
83 1/4	1/16	83 1/4
83 1/2	1/16	83 1/2
83 3/4	1/16	83 3/4
84	1/16	84
84 1/4	1/16	84 1/4
84 1/2	1/16	84 1/2
84 3/4	1/16	84 3/4
85	1/16	85
85 1/4	1/16	85 1/4
85 1/2	1/16	85 1/2
85 3/4	1/16	85 3/4
86	1/16	86
86 1/4	1/16	86 1/4
86 1/2	1/16	86 1/2
86 3/4	1/16	86 3/4
87	1/16	87

# *Tentative Specifications for*

## PHENOLIC LAMINATED SHEET FOR RADIO APPLICATIONS<sup>1</sup>



A.S.T.M. Designation: D 467 - 44 T

ISSUED, 1937; REVISED, 1940, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover phenolic laminated sheet of the following five grades for radio or similar applications where high frequencies are involved.

*Grade No. 1.*—A paper base punching stock for use where dielectric losses are of minor importance,

*Grade No. 2.*—A paper base punching stock having medium dielectric loss,

*Grade No. 3.*—A paper base punching stock having low dielectric loss,

*Grade No. 4.*—A hard paper base stock having low dielectric loss, and

*Grade No. 5.*—A fabric base stock having medium dielectric loss.

NOTE.—Usual applications for the various grades are given in the Appendix.

### Material

2. The material shall consist of layers of paper or cotton fabric as required. These layers shall be suitably impregnated with a phenolic type resinous compound and consolidated into hard

dense sheets under the action of heat and pressure.

### Punching Quality

3. (a) The grades of material differ in their suitability for punching but any of the grades may be punched in simple shapes and thin sizes provided good punching practice is used, including sharp, close clearance dies, proper strip-per plates, and proper heating conditions. When using good punching practice as outlined in Paragraph (c), the various grades shall punch satisfactorily in thicknesses up to and including the maximum limits given in Table I.

(b) Where punching properties better than those listed in Table I are required for particular parts, this shall be subject to agreement between the manufacturer and the purchaser.

(c) In good punching practice, the edges of the piece shall be not closer to the edge of the strip than twice the thickness of the sheet; the holes shall be not smaller in diameter than the thickness of the sheet nor have square corners; the distance between holes or between holes and edge of piece shall be not less

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1944.

than the thickness of the sheet. Heating the material to a temperature of 120 to 140 C. (approximately 15 min. for material  $\frac{1}{8}$  in. in thickness) is generally necessary for best punching results, although in grade No. 1 this may make the material too soft. In this case, better results may be obtained by heating at lower

TABLE I.—MAXIMUM THICKNESS FOR VARIOUS GRADES AT WHICH SATISFACTORY PUNCHING RESULTS MAY BE OBTAINED WHEN USING GOOD PUNCHING PRACTICE.

Grade	At Room Temperature (20 to 30 C.)	When Heated to 120 to 140 C. Before Punching
No. 1.....	$\frac{1}{8}$ in.	$\frac{1}{8}$ in.
No. 2.....	$\frac{3}{8}$ in.	$\frac{3}{8}$ in.
No. 3.....	.....	$\frac{1}{8}$ in.
No. 4.....	.....	$\frac{1}{8}$ in. in simple shapes only
No. 5.....	.....	$\frac{1}{8}$ in.

NOTE.—With simple forms and special precautions greater thicknesses than the above can sometimes be punched. With poor dies, poor punching practice, or intricate parts, good results cannot be expected in the thicknesses listed in the table.

temperatures or for a shorter time. If more than 2 min. elapse between the time the strip leaves the heating medium and the last piece is punched, results will be poor.

### Flexural Strength

4. (a) The flexural strength in either direction of the sheet, tested flatwise, shall be not less than 11,000 psi.

(b) The flexural strength shall be determined in accordance with the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials.<sup>3</sup>

### Power Factor, Dielectric Constant, and Dielectric Loss Factor

5. (a) The values for power factor, dielectric constant, and dielectric loss factor when measured at a frequency of 1,000,000 cycles shall not exceed those prescribed in Table II.

(b) The apparatus and general method of test shall conform to the Tentative

Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup> The sizes of specimens and electrodes shall be as listed in Table III. The test on material

TABLE II.—MAXIMUM PERMISSIBLE VALUES FOR POWER FACTOR, DIELECTRIC CONSTANT, AND DIELECTRIC LOSS FACTOR MEASURED PERPENDICULAR TO LAMINATIONS.

Frequency = 1,000,000 cycles.

Grade	Values on Material as Received			Values on Material After 24 hr. Immersion in Water	
				Thick- nesses $\frac{1}{8}$ in. and over	Thick- nesses under $\frac{1}{8}$ in.
	Power Factor	Di- elec- tric Con- stant	Di- elec- tric Loss Factor	Power Factor	Power Factor
No. 1.....	0.060	...	...	0.100	0.150
No. 2.....	0.045	5.5	0.25	0.050	0.060
No. 3.....	0.030	5.2	0.16	0.035	0.045
No. 4.....	0.035	5.2	0.18	0.040	.....
No. 5.....	0.045	5.5	0.25	0.050	.....

TABLE III.—SIZE OF SPECIMENS AND ELECTRODES FOR MEASUREMENTS OF POWER FACTOR AND DIELECTRIC CONSTANT.

Thickness, in.	Size of Specimen, in.	Diameter of Disk Electrodes, in.
Under $\frac{1}{8}$ .....	6 by 6	3
$\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	6 by 6	4.5
Over $\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	8 by 8	6
Over $\frac{1}{2}$ to $\frac{3}{4}$ , incl.....	10 by 10	8
Over $\frac{3}{4}$ to 1, incl.....	14 by 14	11

as received shall be made on specimens under ordinary laboratory conditions provided the temperature is between 20 and 30 C. The immersion for 24 hr. in water shall be made in a fresh supply of clean tap water held at  $25 \pm 2$  C. For material  $\frac{1}{8}$  in. and under in thickness duplicate specimens from the same sheet shall be used, one for the as-received test and the other for the water-immersion test. For thicker materials, the as-received specimen may be used for water immersion, provided the disk electrode

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



is removed, the grease washed off with benzol or gasoline and the specimen wiped carefully and allowed to air dry for 1 hr. before placing it in the water. In all cases, specimens shall be removed from the water at the end of 24 hr. and the excess water wiped off with a soft cloth; then the electrodes shall be applied and measurements made immediately.

TABLE IV.—PERMISSIBLE VARIATIONS IN SIZE OF PIECES CUT FROM STANDARD SHEETS.

Nominal Thickness, in.	Permissible Variations in Length or Width, plus or minus, in.		
	6 in. and under	over 6 to 24 in.	24 in. and over
0.010 to $\frac{1}{4}$ , incl. ....	0.010	0.015	$\frac{1}{32}$
$\frac{1}{4}$ to $\frac{1}{2}$ , incl. ....	0.012	0.017	$\frac{1}{32}$
$\frac{1}{2}$ to 1, incl. ....	0.015	0.020	$\frac{1}{32}$

TABLE V.—RANGES OF STANDARD THICKNESSES AVAILABLE.

Grade	Range of Standard Thicknesses, in.
No. 1 .....	0.010 to $\frac{1}{4}$ , incl.
No. 2 .....	$\frac{1}{32}$ to $\frac{1}{4}$ , incl.
No. 3 .....	$\frac{1}{32}$ to $\frac{1}{4}$ , incl.
No. 4 .....	$\frac{1}{16}$ to 1, incl.
No. 5 .....	$\frac{1}{16}$ to $\frac{1}{2}$ , incl.

#### Size of Sheet

6. Sheets shall be furnished in manufacturer's standard sheet sizes. The length and width may vary 1 in. over or under the manufacturer's standard size.

NOTE.—In general, manufacturer's standard widths are between 36 and 48 in.

#### Permissible Variations in Size of Small Pieces

7. When small pieces are ordered to be cut from standard size sheets the permissible variations from the specified length or width shall be within the requirements prescribed in Table IV.

#### Standard Thicknesses

8. The nominal thickness of the material shall be specified in the order. The ranges of standard thicknesses for each grade are shown in Table V.

#### Permissible Variations in Thickness

9. The permissible variations from the standard thicknesses of the various grades shall be within the requirements prescribed in Table VI. At least 90 per cent of the area of the sheet shall be within the variations given in Table VI and at no point shall the thickness as measured vary from the nominal by a

TABLE VI.—PERMISSIBLE VARIATIONS IN THICKNESS.

Nominal Thickness, <sup>a</sup> in.		Permissible Variations plus or minus, in.	
Fraction of Inch	Decimal Equivalent	Grades Nos. 1, 2, 3, and 4	Grade No. 5
0.010	0.010 .....	0.002	.....
0.015	0.015 .....	0.0025	.....
0.020	0.020 .....	0.003	.....
0.025	0.025 .....	0.0035	.....
$\frac{1}{32}$	0.031 .....	0.0035	.....
$\frac{1}{64}$	0.047 .....	0.0045	.....
$\frac{1}{16}$	0.0625 .....	0.005	0.0075
$\frac{3}{32}$	0.094 .....	0.007	0.009
$\frac{1}{8}$	0.125 .....	0.008	0.010
$\frac{3}{16}$	0.156 .....	0.009	0.011
$\frac{1}{4}$	0.1875 .....	0.010	0.0125
$\frac{5}{16}$	0.219 .....	0.011	0.014
$\frac{3}{8}$	0.250 .....	0.012	0.015
$\frac{7}{16}$	0.3125 .....	0.0145	0.0175
$\frac{1}{2}$	0.375 .....	0.017	0.020
$\frac{5}{8}$	0.438 .....	0.019	0.022
$\frac{3}{4}$	0.500 .....	0.021	0.024
$\frac{7}{8}$	0.625 .....	0.024	0.027
1	0.750 .....	0.027	0.029
	0.875 .....	0.030	0.031
	1.000 .....	0.033	0.033

<sup>a</sup> On sheet of nominal thickness not listed in the above table, the permissible variations shall be the same as given for the next greater thickness.

value greater than 125 per cent of the specified variation.

#### Color and Finish

10. The color and finish of the material shall be as specified in the order. The standard colors and finishes for the various grades are listed in Table VII.

#### Workmanship

11. The material shall be uniform in quality consistent with the properties prescribed in these specifications. It shall be free from blisters, wrinkles or cracks and reasonably free from other small defects such as scratches, dents, solvent areas, etc.

**Packing**

12. Unless otherwise specified, material shall be packed in substantial crates or boxes in accordance with the usual practice of the individual manufacturer.

**Marking**

13. Every full-size sheet shall be marked with the manufacturer's name or trade-mark, and the A.S.T.M. grade

TABLE VII.—STANDARD COLORS AND FINISHES AVAILABLE.

Grade	Color <sup>a</sup>	Finish
No. 1 ...	Natural, Black, Chocolate	Semigloss or Dull
No. 2 ....	Natural, Black, Chocolate	Semigloss or Dull
No. 3 ....	Natural only	Semigloss or Dull
No. 4 ....	Natural or Black	Polished or Semigloss
No. 5 ....	Natural only	Semigloss

<sup>a</sup> *Natural* is the color produced by the natural undyed paper or fabric and resin used and may vary from a light tan to a light brown or reddish brown. This natural color shall be free from streaks or stains, and substantially uniform except for grades Nos. 2 and 3 which may be mottled in appearance.

*Black* sheets shall have substantially uniform black surfaces and black body. A slight grayish-black color on sawed or machined edges is permissible.

*Chocolate* sheets shall have a uniform dark brown or chocolate surface with natural core.

number and designation D 467. The containers shall be marked with the manufacturer's name, A.S.T.M. grade number and designation D 467, size and quantity of sheets therein, together with other marking required by the purchaser.

**Inspection**

14. (a) The manufacturer shall afford the inspector representing the purchaser all reasonable facilities, without charge, to satisfy him that the material is being

furnished in accordance with these specifications. Tests and inspection at the place of manufacture may be made prior to shipment, and such inspection shall be final. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

(b) In case material is not inspected at the place of manufacture, the purchaser may make the tests to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

**Rejection**

15. (a) Unless otherwise specified, any rejections based on tests made in accordance with Section 14 (b) shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

(b) Any portion of an accepted shipment of material which is subsequently found not to be in accordance with these specifications, will be rejected, provided the manufacturer is notified within 90 days from the date of receipt of the material by the purchaser.

(c) Samples tested in accordance with Section 14 (b) that represent rejected material, shall be preserved for one month from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

**APPENDIX****USUAL APPLICATIONS FOR THE VARIOUS GRADES OF PHENOLIC LAMINATED SHEETS**

*Grade No. 1.*—Sockets, terminal boards, miscellaneous instrument parts where insulating properties and moisture resistance are of minor importance and good punching and assembling characteristics essential.

*Grade No. 2.*—General use for insulation in radio frequency circuits where punching and assembling operations are not particularly severe.

*Grade No. 3.*—Simple punched parts where very low losses are essential, such as variable

capacitor insulation and metal tube insulation and some range switch parts.

*Grade No. 4.*—General use in instruments and other applications where parts are produced by machining and where reasonably good appearance and good electrical properties are desired.

*Grade No. 5.*—Applications similar to Grade No. 2 except for more severe punching and assembling operations.



# *Tentative Specifications for*

## ROUND PHENOLIC LAMINATED TUBING FOR RADIO APPLICATIONS<sup>1</sup>



A.S.T.M. Designation: D 616 - 41 T

ISSUED, 1941.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover round phenolic laminated tubing for radio and similar applications where high frequencies are involved. Either rolled or molded tubes may be furnished in accordance with these specifications provided they conform to the prescribed requirements. The grade of tubes shall be specified in the order. In general, molded tubes are somewhat more dense and have slightly better moisture resistance than rolled tubes, but have mold seams which are a source of weakness both mechanically and electrically, particularly in thin-walled tubes. Where molded tubes are threaded, the threads may be poor at the seams. The following grades are covered:

*Grade No. T-1.*—A strong, tough paper base tube in which dielectric losses are of minor importance, and

*Grade No. T-2.*—A paper base tube with low dielectric loss and excellent

threading characteristics, but not quite as tough as grade T-1.

NOTE.—Usual applications for these grades are given in the Appendix.

### Material

2. The material shall consist of layers of paper suitably impregnated with a phenolic type resinous compound and formed into tubular shape, these layers being subsequently consolidated under action of heat and pressure into a hard dense tube which shall then be ground to size. Rolled tubes shall be cured by baking in ovens after rolling and molded tubes shall be cured in molds under pressure.

### Punching Quality

3. (a) Grade T-2 is not quite as suitable for punching as grade T-1, but either grade may be punched in simple shapes and thin sizes provided good punching practice (Note) sharp, close-clearance dies, proper strippers, and proper heating conditions are used.

(b) The maximum wall thickness at which satisfactory punching results may

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1941.

be obtained when using good punching practice is  $\frac{1}{16}$  in.

NOTE.—In good punching practice, wherever there is more than one row of holes parallel with the axis of the tube, the inside punching mandrel, pin, or horn shall not be smaller in diameter than 0.005 in. less than the minimum inside diameter of the size tube being punched, using the prescribed permissible variations in diameter for the grade; the holes shall not be smaller in diameter than the wall thickness nor have square corners; the distance between holes, or between holes and end or edge of tube shall not be less than the wall thickness. Heating the material to a temperature of 120 to 140 C. (the heating period will vary and should not exceed 15 min. for material up to  $\frac{3}{8}$  in. in thickness) is generally necessary for best punching results. If more than 2 min. elapse between the time the tube leaves the heating medium and punching is completed, the results will be poor. An interval of not over 30 sec. is preferable.

#### Axial Compressive Strength

4. The compressive strength of the tubing tested axially, with load applied perpendicular to the faces or ends of the specimen, shall not be less than the following:

Grade	Axial Compressive Strength, min., psi.
No. T-1 .....	10 000
No. T-2 .....	12 000

#### Density of Rolled Tubing

5. Since density is a measure of the uniformity of rolled tubing, in no case shall the density be less than the following:

Grade	Density, min., g. per cu. cm.
No. T-1 .....	1.10
No. T-2 .....	1.15

#### Power Factor, Dielectric Constant, and Dielectric Loss Factor

6. The values for power factor, dielectric constant, and dielectric loss factor when measured at a frequency of 1,000,000 cycles shall not exceed those prescribed in Table I.

TABLE I.—MAXIMUM PERMISSIBLE VALUES FOR POWER FACTOR, DIELECTRIC CONSTANT, AND DIELECTRIC LOSS FACTOR MEASURED PERPENDICULAR TO LAMINATIONS.

Frequency = 1,000,000 cycles.

Grade	Values on Material as Received			Values on Material After 24-hr. Immersion in Water	
				Wall Thicknesses $\frac{3}{4}$ in. and over	Wall Thicknesses $\frac{3}{8}$ to $\frac{3}{4}$ in., excl.
	Power Factor	Dielectric Constant	Dielectric Loss Factor	Power Factor	Power Factor
No. T-1 .....	0.040	5.5	0.22	0.070	0.12
No. T-2 .....	0.040	5.0	0.20	0.055	0.08

#### Length

7. The lengths of tubing shall be as specified in the order and shall be furnished either in standard lengths or cut to size, as follows:

(a) *Standard Lengths.*—Manufacturer's standard lengths are 18 to 24 in. for tubing less than  $\frac{1}{4}$  in. in inside diameter, and 36 to 48 in. for tubing of larger diameters, unless otherwise specified.

(b) *Cut to Size.*—When small pieces of tubing are ordered cut to size, the permissible variations in length shall be within the requirements prescribed in Table II.

TABLE II.—PERMISSIBLE VARIATIONS IN LENGTHS OF TUBING CUT TO SIZE.

Nominal Length, in.	Permissible Variations, plus or minus, in.		
	$\frac{3}{8}$ to 2 in. in Outside Diameter	Over 2 to 4 in. in Outside Diam.	Over 4 in. in Outside Diameter
0 to 3 .....	0.010	0.010	0.030
Over 3 to 6 .....	0.010	0.015	0.030
Over 6 to 12 .....	0.015	0.020	0.030
Over 12 to 48 .....	0.030	0.030	0.050

#### Standard Sizes, Inside and Outside Diameter

8. (a) The nominal inside and outside diameter shall be specified in the order.

The standard ranges of available diameters for both grades T-1 and T-2 are shown in Table III.

TABLE III.—RANGES OF STANDARD INSIDE AND OUTSIDE DIAMETERS AVAILABLE.

Nominal Wall Thickness, in.	Inside Diameter, in.	Outside Diameter, in.
Rolled tubes, $\frac{1}{16}$ " to $\frac{1}{2}$ "....	$\frac{1}{8}$ to 12	$\frac{3}{16}$ to 12½
Molded tubes, $\frac{1}{16}$ to $\frac{1}{2}$ "....	$\frac{1}{8}$ to 3½	$\frac{1}{4}$ to 4

<sup>a</sup> While rolled tubes in small diameters are available in wall thicknesses as small as 0.010 in., the properties and dimensions prescribed in these specifications apply only to tubes having wall thicknesses of  $\frac{1}{16}$  in. and over.

(b) The standard steps in inside and outside diameters available are shown in Table IV.

TABLE IV.—STANDARD STEPS IN INSIDE AND OUTSIDE DIAMETERS.

Nominal Diameter, in.	Diameters Available in Steps of, in.
$\frac{1}{8}$ to $\frac{1}{2}$ .....	$\frac{1}{64}$
Over $\frac{1}{2}$ to $1\frac{1}{2}$ .....	$\frac{1}{32}$
Over $1\frac{1}{2}$ to 3.....	$\frac{1}{16}$
Over 3 to 6.....	$\frac{1}{8}$
Over 6 to 8.....	$\frac{1}{4}$
Over 8 to 12.....	$\frac{1}{2}$

### Permissible Variations in Diameter

9. The permissible variations in inside and outside diameter shall be within the requirements prescribed in Table V.

TABLE V.—PERMISSIBLE VARIATIONS IN INSIDE AND OUTSIDE DIAMETER.

Nominal Inside and Outside Diameters, in.	Permissible Variations, plus or minus, in.	
	Inside Diameter	Outside Diameter
$\frac{1}{8}$ to $\frac{3}{16}$ .....	0.003	0.005
$\frac{3}{16}$ to $1\frac{1}{8}$ .....	0.004	0.005
2 to 4.....	0.008	0.008
4½ to 12½ (rolled tubes only).....	0.010	0.025

### Permissible Variations in Wall Thickness

10. The permissible variations in wall thickness shall be within the requirements prescribed in Table VI. The

wall thickness shall be measured with a tube or ball foot micrometer.

TABLE VI.—PERMISSIBLE VARIATIONS IN WALL THICKNESS.

Nominal Wall Thickness, in.	Permissible Variations, plus or minus, in.			
	Rolled Tubing	Molded Tubing		
	All Inside Diameters	$\frac{1}{8}$ to $\frac{1}{2}$ in. in Inside Diameter	Over $\frac{1}{2}$ to $\frac{3}{4}$ in. in Inside Diameter	Over $\frac{3}{4}$ in. in Inside Diameter
$\frac{1}{16}$ and under.....	0.006	0.008	0.008	0.008
$\frac{1}{16}$ to $\frac{1}{8}$ , excl.....	0.007	0.011	0.011	0.011
$\frac{1}{8}$ to $\frac{1}{4}$ , excl.....	0.009	0.017	0.015	0.011
$\frac{1}{4}$ to $\frac{1}{2}$ , incl.....	0.011	0.025	0.018	0.013

### Color and Finish

11. The color and finish shall be as specified in the order. The standard colors and finishes are listed in Table VII.

TABLE VII.—STANDARD COLORS AND FINISHES

Grade	Color <sup>a</sup>	Finish <sup>b</sup>
No. T-1.....	Natural or Black	Ground or Buffed
No. T-2.....	Natural or Black	Ground or Buffed

<sup>a</sup> *Natural* is the color produced by the natural undyed paper and the resin used, and may vary from a light tan to a light brown or reddish brown.

*Black* is the color produced by natural undyed paper and a black dyed resin and will be grayish black in color, particularly on sawed or machined surfaces, or where layers may be partially cut through in grinding tube to diameter.

<sup>b</sup> *Ground finish* is that finish applied by a fine grinding wheel or belt, is free from any pronounced scratches, and is suitable for majority of applications.

*Buffed finish* is somewhat more glossy than the ground finish, and is obtained by buffing the tube following grinding using a touch of shellac or other polishing compound on the buffer.

*Varnished finish* is a third finish that is sometimes applied to tubes for special decorative or chemical resistant applications. It is obtained by coating the tube one or more times with a varnish or lacquer and air-drying or baking. Because the varnished finish requires operations which may change greatly the physical and electrical properties, such finished tubes are not covered by these specifications.

### Methods of Testing

12. The properties enumerated in these specifications shall be determined in accordance with the following methods of test of the American Society for Testing Materials:

(a) *Axial Compressive Strength and Density*.—Standard Methods of Testing Laminated Tubes Used in Electrical

Insulation (A.S.T.M. Designation: D 348).<sup>3</sup>

(b) *Power Factor, Dielectric Constant, and Dielectric Loss Factor.*—The apparatus and general method of test shall conform to the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup> The sizes of specimens and electrodes shall be as listed in Table VIII. The test on material as received shall be

TABLE VIII.—SIZES OF SPECIMENS AND ELECTRODES FOR TUBING.

Nominal Wall Thickness, in.	Inside Diameter of Specimen, min., in.	Length <sup>a</sup> of Specimen, in.	Length <sup>b</sup> of Electrodes, in.
$\frac{1}{8}$ and under.....	$\frac{1}{2}$	5	4
Over $\frac{1}{8}$ to $\frac{1}{4}$ , incl.....	1	5	4
Over $\frac{1}{4}$ to $\frac{1}{2}$ incl.....	2	6	4

<sup>a</sup> If tubes of smaller diameter or greater wall thickness than those listed in Table IV are to be tested, electrodes of sufficient area to have a capacity of approximately 100  $\mu\text{f.}$  shall be used and the tubes shall extend at each end beyond the electrodes for a length of  $\frac{1}{2}$  in. up to five times wall thickness.

<sup>b</sup> The electrodes shall be of equal length and shall be in line with each other.

made on specimens under ordinary laboratory conditions provided the temperature is between 20 and 30 C. The immersion for 24 hr. in water shall be made in a fresh supply of clean tap water held at  $25 \pm 2$  C. For material  $\frac{1}{4}$  in. and under in wall thickness duplicate specimens from the same length of tubing shall be used, one for the as-received test and the other for the water-immersion test. For thicker materials, the as-received specimen may be used for water immersion, provided the lead for the electrode is removed, the grease washed off with toluol or gasoline and the specimen wiped carefully and allowed to air dry for 1 hr. before placing it in the water. In all cases, specimens shall be removed from the water at the end of 24 hr. and the excess water wiped off

with a soft cloth; then the electrodes shall be applied and measurements made immediately.

### Workmanship

13. The material shall be uniform in quality, consistent with the properties prescribed in these specifications. Tubes shall be free from metal particles, blisters, wrinkles, cracks, pronounced rings, flat spots, loose inside laps, or pronounced pits or scratches on the inside.

### Packing

14. Unless otherwise specified, the tubing shall be packed in substantial boxes in accordance with the usual practice of the individual manufacturer.

### Marking

15. The containers shall be marked with the manufacturer's name, grade, A.S.T.M. Designation D 616, size and quantity of tubing therein, together with other marking required by the purchaser.

### Inspection

16. (a) The manufacturer shall afford the inspector representing the purchaser, all reasonable facilities, without charge, to satisfy him that the material is being furnished in accordance with these specifications. Tests and inspection at the place of manufacture may be made prior to shipment, and such inspection shall be final. All tests and inspection shall be so conducted as not to interfere unnecessarily with the operation of the works.

(b) In case material is not inspected at the place of manufacture, the purchaser may make the tests to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests, however, shall be made at the expense of the purchaser.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Rejection

17. (a) Unless otherwise specified, any rejections based on tests made in accordance with Section 16 (b) shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

(b) Any portion of an accepted shipment of material which is subsequently found not to be in accordance with these

specifications, will be rejected, provided the manufacturer is notified within 90 days from the date of receipt of the material by the purchaser.

(c) Samples tested in accordance with Section 16 (b) that represent rejected material, shall be preserved for one month from the date of the test report. In case of dissatisfaction with the results of the tests, the manufacturer may make claim for a rehearing within that time.

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## APPENDIX

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### USUAL APPLICATIONS FOR THE VARIOUS GRADES OF PHENOLIC LAMINATED TUBING

*Grade T-1.*—Coil forms in receiving sets and other applications where good punching characteristics are essential and where the insulating properties under moist conditions need not be quite as good as those of grade T-2.

*Grade T-2.*—General use in transmitting coil forms and in certain radio frequency apparatus where low losses are essential even under humid conditions.

# *Tentative Methods of* MEASURING DIMENSIONS OF RIGID RODS USED IN ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 741 - 43 T

ISSUED, 1943.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods cover procedures for measuring the dimensions of all rigid rods used in electrical insulation, the limitations imposed being those of the size range of the more common forms of measuring instruments used in making the test.

## Conditioning

2. In cases of disagreement between the seller and the purchaser because of dimensional changes resulting from changes in temperature and humidity, all specimens shall be conditioned for 48 hr. at  $25 \pm 1$  C. and  $50 \pm 2$  per cent relative humidity, and measured under these conditions. Measurements by both interested parties shall be made within a three-week period

## Method of Measurement

3. All measurements shall be made in accordance with the procedures described in the Standard Methods of Test for Thickness of Solid Electrical Insulation

(A.S.T.M. Designation: D 374)<sup>3</sup>, as applicable. The micrometer or vernier calipers shall be closed slowly on the specimen until contact is made without appreciable distortion of the specimen. The criterion of contact is the initial development of frictional resistance to movement of the specimen between the micrometer or caliper surfaces.

## Length of Rods 12 in. and Under in Length

4. (a) *Apparatus*.—A standard machinist's micrometer or vernier calipers of suitable size reading to 1 mil (0.001 in.) shall be used.

(b) *Test Specimens*.—Specimens shall consist of rods cut to lengths of 12 in. and under.

(c) *Procedure*.—The length of the specimen shall be measured to the nearest 0.001 in. Size permitting, four measurements shall be made at points 90 deg. apart around the circumference of the specimen.

(d) *Report*.—The average of the measurements taken shall be reported as the length of the rod.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, August 30, 1943.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

**Length of Rods over 12 in. in Length**

5. (a) *Apparatus*.—A machinist's precision steel tape or steel scale of suitable length graduated in  $\frac{1}{64}$  in. shall be used.

(b) *Test Specimens*.—Specimens shall consist of rods over 12 in. in length, including full lengths.

(c) *Procedure*.—The length of the specimen shall be measured to the nearest  $\frac{1}{64}$  in. Size permitting, four measurements shall be made at points 90 deg. apart around the circumference of the specimen.

(d) *Report*.—The average of the measurements taken shall be reported as the length of the rod.

**Diameter**

6. (a) *Apparatus*.—A standard machinist's micrometer or vernier calipers reading to 1 mil (0.001 in.) shall be used.

(b) *Test Specimens*.—Specimens shall consist of rods of any diameter.

(c) *Procedure*.—The diameter of the specimen shall be measured to the nearest 0.001 in. Size permitting, four measurements shall be made at points 45 deg. apart around the circumference at each end and at the middle of the specimen.

(d) *Report*.—The average of the twelve measurements taken shall be reported as the diameter of the rod.

**Warp**

7. (a) *Apparatus*.—A horizontal flat surface and a rigid bar with a vertically plane surface firmly fixed at right angles to the flat surface both being at least as long as the specimen shall be used. The height of the bar shall exceed half the diameter of the rod. Feeler gages

of suitable thicknesses will also be required.

(b) *Test Specimens*.—Specimens shall consist of rods of any length or diameter.

(c) *Procedure*.—The specimen shall be placed on the horizontal flat surface and rotated against the vertically plane surface of the rigid bar. The bar shall be firmly fastened to the horizontal flat surface. With the aid of feeler gages, the maximum distance of the nearest point of the rod from the vertically plane surface shall be measured to the nearest 0.001 in.

(d) *Report and Calculations*.—Warp or lack of straightness shall be reported as the maximum distance of any part of the rod from a straight edge which connects the ends of the specimen. The warp shall be calculated as follows:

$$W = \frac{D \times 100}{L}$$

where:

$W$  = percentage of warp,

$D$  = maximum deviation of rod from straight edge in inches, and

$L$  = length of rod in inches as determined in Sections 4 or 5.

In order to compare the relative warpage for any length of rod, the warp shall be calculated as the percentage warp for a 36-in. length as follows:

$$C = \frac{W \times 36}{L}$$

where:

$C$  = percentage of warp calculated for 36-in. length,

$W$  = percentage of warp, and

$L$  = length of rod in inches as determined in Sections 4 or 5.

# *Tentative Methods of*

## TESTING NONRIGID POLYVINYL TUBING<sup>1</sup>



A.S.T.M. Designation: D 876 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover procedures for the testing of nonrigid polyvinyl chloride tubing, or its copolymers with vinyl acetate, used as electrical insulation. For the purpose of these methods nonrigid tubing shall be tubing having an initial elongation in excess of 100 per cent at break.

### Sampling

2. (a) A sufficient number of pieces of tubing shall be selected in such a manner as to be representative of the shipment.

(b) The number of specimens required for the purpose of tests shall be cut from the pieces selected in accordance with Paragraph (a), and care shall be taken to select material which is free from obvious defects.

### Test Conditions

3. Unless otherwise specified in these methods, tests shall be conducted at

atmospheric pressure and at a temperature of  $25 \pm 1$  C. ( $77 \pm 1.8$  F.). Room temperature as stated in these methods shall be within this temperature range.

### DIMENSIONAL TESTS

#### Apparatus

4. The apparatus shall consist of the following:

(a) *Tapered-Steel Gages*.—The gages shall be chromium-plated and suitable for covering the range of tubing sizes shown in Table I. The gages shall have a uniform taper of 0.010 in. in diameter per 1 in. of length, and shall be graduated with circular lathe-cut rings every 0.5 in. of length. The graduations shall then represent a uniform increase in diameter of 0.005 in. in diameter per 0.5 in. of length.

(b) *Micrometers*.—The micrometers shall be of the machinist's type and suitable for covering the range of tubing sizes shown in Table I.

(c) *Steel Scale*.—A steel scale graduated in 0.01 in.

#### Test Specimens

5. A 1-in. specimen free of kinks shall

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.



be cut from the sample. The cutting shall be done perpendicular to the longitudinal axis of the tubing specimen, and shall result in a specimen 1 in. in length having cleanly cut square edges.

### Procedure for Measuring Inside Diameter

6. (a) A gage which will fit part way into the specimen shall be selected. The

TABLE I.—SIZES OF NONRIGID POLYVINYL CHLORIDE TUBING, OR ITS COPOLYMERS WITH VINYL ACETATE.

Size	Inside Diameter, in		
	Maximum	Minimum	Nominal
2 in. ....	2.070	2.000	....
1½ in. ....	1.812	1.750	....
1¼ in. ....	1.550	1.500	....
1½ in. ....	1.290	1.250	....
1 in. ....	1.036	1.000	....
¾ in. ....	0.911	0.875	....
¾ in. ....	0.786	0.750	....
¾ in. ....	0.655	0.625	....
¾ in. ....	0.524	0.500	....
¾ in. ....	0.462	0.438	....
¾ in. ....	0.399	0.375	....
¾ in. ....	0.334	0.3125	....
No. 0. ....	0.347	0.325	0.330
No. 1. ....	0.311	0.289	0.294
No. 2. ....	0.278	0.258	0.263
No. 3. ....	0.249	0.229	0.234
No. 4. ....	0.224	0.204	0.208
No. 5. ....	0.198	0.182	0.186
No. 6. ....	0.178	0.162	0.166
No. 7. ....	0.158	0.144	0.148
No. 8. ....	0.141	0.129	0.133
No. 9. ....	0.124	0.114	0.118
No. 10. ....	0.112	0.102	0.106
No. 11. ....	0.101	0.091	0.095
No. 12. ....	0.089	0.081	0.085
No. 14. ....	0.072	0.064	0.066
No. 16. ....	0.061	0.051	0.053
No. 18. ....	0.049	0.040	0.042
No. 20. ....	0.039	0.032	0.034

specimen shall be slipped, without forcing (Note), over the gage until there is no visible air space between the end of the specimen and the gage anywhere on the circumference. This point on the gage shall be considered the inside diameter of the specimen.

NOTE.—When the tubing specimen tends to stick, the gage may be dipped in water to facilitate slipping the specimen over the gage. However, when water is used as a lubricant on the gage, great caution should be exercised to make certain that the specimen is not forced on the gage, thereby stretching the specimen.

(b) The diameter at the point of contact between the specimen and gage shall be determined by referring to the nearest visible graduation. Any distance between the edge of the specimen and the nearest graduation shall be measured with the steel scale. Each 0.1 in. on the length of the gage represents an increase of 0.001 in. in diameter. Since the diameter at the nearest graduation is known, the inside diameter of the specimen shall be obtained by interpolation and reported to the nearest 0.001 in.

### Procedure for Measuring Outside Diameter

7. The specimen shall be measured for outside diameter when located on the tapered gage as described in Section 6. Three measurements approximately 120 deg. apart shall be made adjacent to the edge of each specimen. The measurements shall be made in accordance with the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374),<sup>3</sup> using Method B, and the following additional details shall be observed:

(1) The micrometer shall be supported to allow both hands to be free for manipulation.

(2) The outside diameter shall be measured adjacent to, but not on or over the cut edge.

(3) One side of the specimen shall be held lightly against the fixed anvil of the micrometer and shall be slowly oscillated back and forth. The rotating anvil shall then be moved up very slowly until the first definite increase in resistance to the movement of the specimen is felt.

### Report

8. The report shall include the following:

(1) The inside diameter of the specimen to the nearest 0.001 in.,

<sup>3</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

- (2) All readings on outside diameter, of the specimen to the nearest 0.001 in.
- (3) The average outside diameter, and
- (4) The average wall thickness.

#### FLAMMABILITY TEST

NOTE.—This test is not a measure of the flammability of the compound but is a measure of the flammability of a given tubing with an indicated inside diameter and wall thickness.

#### Apparatus

9. The apparatus shall consist of the following:

(a) *Sheet Metal Frame*.—A three-walled sheet metal frame 12 in. in width 14 in. in depth and 29 in. in height. The walls facing each other shall support two parallel horizontal steel rods, which shall be separated 16 to 21 in. as required, and with the lower rod closer to the remaining (back) wall, located so that they define a plane which shall be at an angle of 70 deg. with the horizontal.

(b) *Bare Steel Wire*.—A length of bare steel wire, approximately 0.029 in. in diameter for all sizes of specimens, shall be attached at one end to the center of the upper rod. The other end of the wire shall pass over the center of the lower rod and shall have a 1-lb. weight attached to maintain the upper rod in such a position that the wire is in a straight line across the two rods.

(c) *Tirrill Burner*.—A Tirrill burner having a nominal bore of  $\frac{3}{8}$  in. and a length of approximately 4 in. above the primary inlets, with an attached pilot light.

(d) *Gas Supply*.—A supply of ordinary illuminating gas at normal pressure.

(e) *Time Piece*.—A time piece measuring seconds.

(f) *Flame Indicators*.—Strips of gummed paper.

#### Test Specimens

10. For tubing sizes  $\frac{1}{8}$  in. and under in

inside diameter, five test specimens 17 in. in length shall be cut from the sample. For tubing sizes 1 in. and over in inside diameter, five test specimens 22 in. in length shall be cut from the sample.

#### Procedure

11. (a) The test shall be made in a room or cabinet free from drafts of air. The specimen shall be drawn onto the wire. The covered wire shall be fastened into position, with its length adjusted so that the weight hangs free, maintaining tension and supporting the specimen in a straight line at an angle of 70 deg. with the horizontal. One end of the specimen shall be near the lower rod, and the other end shall be drawn to the upper rod and clamped so that the specimen is closed, in order to prevent a draft through the tube. The paper indicator shall be applied to the upper end of the specimen, in a position such that, for tubing  $\frac{1}{8}$  in. and under in inside diameter, the lower edge is 10 in. away from the point at which the inner cone of a test flame shall be applied. For tubing 1 in. and over in inside diameter the lower edge of the paper indicator shall be 15 in. away from the point at which the inner cone of the test flame is to be applied. The indicator shall be wrapped and pasted around the specimen.

(b) The height of the flame with the burner in a vertical position shall be adjusted to 5 in., with an inner cone  $1\frac{1}{2}$  in. in height. The burner, with only the pilot lighted, on a base at an angle of 25 deg. from the horizontal, shall be placed in position under the specimen and adjusted so that there is a distance of  $1\frac{1}{2}$  in. between the tip of the stem and the surface of the specimen (along the axis of the burner stem). The valve controlling the gas supply to the burner shall be opened, and the flame applied to the specimen for a period of 15 sec.

(c) When the tubing is flat, the wire shall support the specimen by one of the two small curvatures found in flat tubing. The flats of the specimen shall be directed vertically downward. The distance of  $1\frac{1}{2}$  in. from burner to specimen shall be between the tip of the stem and the surface of the narrow side of the flat specimen. The valve controlling the gas supply to the burner shall be opened, and the flame applied to the narrow side of the specimen for a period of 15 sec.

(d) The duration of burning of the specimen shall be immediately determined after removal of the flame.

(e) The length of specimen burned shall be determined by subtracting the length of the unburned portion (Note) from 10 in. for tubing sizes  $\frac{7}{8}$  in. and under in inside diameter, and from 15 in. for sizes 1 in. and over in inside diameter.

NOTE.—Length of the unburned portion shall be obtained by measuring in inches the distance from the lower edge of the indicator paper to the first obvious defect caused by the flame.

### Report

12. The report shall include the following:

(1) Inside diameter and average wall thickness of the sample from which specimens were taken,

(2) Maximum and minimum durations of burning of the five specimens, and the average duration of burning of the remaining three, and

(3) Maximum and minimum lengths of the five specimens burned, and the average length of specimen burned for the remaining three.

### TENSION TEST

#### Procedure

13. Tensile strength and ultimate elongation shall be determined in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber

(A.S.T.M. Designation: D 412)<sup>3</sup> with the following exceptions:

(1) For sizes Nos. 20 to 0, inclusive, six test specimens shall be prepared by cutting lengths from the sample and subjecting them to the tension test in tubing form.

(2) For sizes  $\frac{5}{8}$  in. to 2 in., inclusive, in inside diameter six test specimens taken from the sample shall be prepared in the form as represented by die B. This shall be accomplished by cutting one wall along a longitudinal axis, flattening the piece and applying die B parallel to this axis.

(3) The inside and outside diameters shall be measured in accordance with Sections 4 to 8.

(4) In determining the tensile strength the average area of the specimens selected shall be used.

(5) Two parallel gage lines for use in determining elongation, shall be marked on the tubing, perpendicular to the longitudinal axis, one on each side of the center and 1 in. therefrom.

(6) The distance between grips of the testing machine shall be 4 in.

(7) The rate of travel of the power actuated grip shall be 12 in. per min., and shall be uniform at all times.

(8) Results on specimens which break outside of the gage marks shall be discarded and retests shall be made.

### Report

14. The report shall include the following:

(1) Size of tubing from which the specimens were taken,

(2) All observed and recorded data on which the calculations are based,

(3) Average tensile strength determined on the best five out of six specimens, and

(4) Average ultimate elongation determined on the best five out of six specimens.

### RESISTANCE TO HEAT AGING TEST

NOTE.—Both of the following Methods A and B should be conducted to obtain full data on resistance to heat aging. Method A should be correlated with the lengthwise shrinkage test (Sections 29 to 33, inclusive), since percentage change in ultimate elongation indicates the resistance of a specimen to aging, provided it has a minimum of internal strains originally. Specimens with initially high internal strains will, in general, show less change in ultimate elongation than those with a minimum of strains.

#### *Method A, Using Tension Test*

##### Apparatus

15. The apparatus shall consist of the following:

(a) *Oven*.—The oven shall conform to the requirements prescribed in Section 4 of the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573).<sup>3</sup>

(b) *Tension Testing Machine*.—The tension testing machine shall be the same as prescribed in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412).<sup>3</sup>

##### Test Specimens

16. Six specimens shall be cut from the sample (Section 25) and shall be prepared in a similar manner to that described in Section 13, items (1) and (2), according to the various sizes of tubing.

##### Procedure

17. (a) Three test specimens shall be suspended vertically in the oven, without touching each other or the sides of the oven. Tubing specimens shall be kept open throughout their entire lengths. The specimens shall be maintained at  $100 \pm 2$  C. for a period of 72 hr., unless otherwise specified. At the end of the specified time, the specimens shall be removed, and kept at room temperature for a period of 16 hr. but

not longer than 20 hr. After the rest period, gage lines 2 in. apart, shall be placed on each specimen, each specimen placed in the tension testing machine and stretched to ultimate elongation as described in Section 13.

(b) Gage lines 2 in. apart shall be placed on each of the remaining three untreated specimens, each specimen placed in the tension testing machine and stretched to ultimate elongation (Note).

NOTE.—The results for elongation obtained in Section 13 may be used for the unaged values.

(c) The ultimate elongations of the aged specimens shall be compared to the ultimate elongations of the unaged specimens. If the ultimate elongations fail to check within 10 per cent of the highest value obtained in the unaged specimens, three additional specimens shall be tested. The final value of ultimate elongation for aged specimens shall be the average of all tests run.

##### Report

18. The report shall include the following:

- (1) The sample size from which specimens were taken,
- (2) Average ultimate elongation of specimens before aging,
- (3) Average ultimate elongation of specimens after aging, and
- (4) Average percentage change in ultimate elongation.

#### *Method B, Using Weight Loss on Heating*

##### Apparatus

19. The apparatus shall consist of the following:

(a) *Chemical Balance*.

(b) *Oven*.—The oven shall conform to the requirements prescribed in Section 4 of the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573).<sup>3</sup>

(c) *Dessicator*,

### Test Specimens

20. Test specimens in full section 6 in. in length shall be cut from the tubing.

### Procedure

21. The specimens shall be placed in a desiccator and conditioned at room temperature over calcium chloride for 24 hr. At the end of this period the specimens shall be immediately weighed, and then suspended vertically in the oven described in Section 19 (b), without touching each other or the sides of the oven. The tubing specimens shall be kept open throughout their entire lengths. The specimens shall be kept at  $100 \pm 2$  C. for a period of 72 hr., unless otherwise specified. At the end of the specified time, the specimens shall be removed, and kept at room temperature over calcium chloride for 1 hr. Upon removal from the desiccator the specimens shall be immediately weighed.

### Report

22. The report shall include the following:

- (1) The sample size from which specimens were taken, and
- (2) The loss of weight calculated as a percentage of the original weight.

### OIL RESISTANCE TEST

NOTE.—The oil resistance test should be correlated with the lengthwise shrinkage test (Sections 29 to 33, inclusive) since percentage change in ultimate elongation indicates the oil resistance of a specimen, provided it has a minimum of internal strains originally. Specimens with initially high internal strains will, in general, show less change in ultimate elongation than those with a minimum of strain.

### Significance of Test

23. The tubing covered in these methods is often used in places where it comes in contact with lubricating oils. While the tubing is in service, the oils may be accidentally spilled on the surface, or

deposited by oil splashed resulting from lubricated moving parts. As a consequence it is important to ascertain the effect of lubricating oil in contact with flexible vinyl tubing, consisting of vinyl chloride, or its copolymers with vinyl acetate.

### Apparatus

24. The apparatus shall be the same as that described in Section 15.

### Test Specimens

25. Three additional specimens shall be selected from the sample used in Section 16.

### Procedure

26. (a) The test specimens shall be totally immersed in A.S.T.M. No. 3 high-swelling oil (Note) as described in the Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (A.S.T.M. Designation: D 471)<sup>3</sup> at a temperature of  $70 \pm 2$  C. for a period of 4 hr., unless otherwise specified. At the end of this time, the specimens shall be removed from the oil, blotted to remove excess oil, allowed to cool at room temperature for 30 min., bathed in mineral spirits at room temperature to remove the remaining film of oil from the surface, and wiped dry. Gage marks 2 in. apart shall be placed on each specimen and the ultimate elongation of each determined.

NOTE.—No. 3 high-swelling oil is identified by the following properties:

Saybolt Universal viscosity,  $155 \pm 5$  sec. at 100 F.

Aniline point,  $69.5 \pm 1$  C.

Flash point,  $330 \pm 5$  F.

(b) The ultimate elongations of the oil-immersed specimens shall be compared with the ultimate elongations of the specimens tested in Section 13. If the ultimate elongations fail to check within 10 per cent of the highest value

obtained for the specimens of Section 13, three additional specimens shall be immersed in oil and tested. The final value of ultimate elongation for specimens immersed in oil shall be the average of all tests run.

### Report

27. The report shall include the following:

- (1) Sample size from which the specimens were taken,
- (2) Average ultimate elongation of the specimens before aging,
- (3) Average ultimate elongation of the specimens after aging, and
- (4) Average percentage change in ultimate elongation.

### BRITTLENESS TEST

#### Procedure

28. The brittleness shall be determined in accordance with the Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746),<sup>3</sup> except as specified in Paragraphs (a) and (b):

(a) For tubing sizes Nos. 20 to 0, inclusive, test specimens in full section  $1\frac{1}{2}$  in. in length shall be cut from the sample.

(b) For tubing  $\frac{5}{16}$  in. to 2 in., inclusive, in inside diameter test specimens  $\frac{1}{4}$  in. in width and  $1\frac{1}{2}$  in. in length shall be cut from the sample. This shall be accomplished by cutting a  $\frac{1}{4}$ -in. strip along a longitudinal axis of the sample.

### LENGTHWISE SHRINKAGE TEST

#### Apparatus

29. The apparatus shall consist of the following:

(a) *Oven*.—The oven shall conform to the requirements prescribed in Section 4 of A.S.T.M. Method D 573.

(b) *Scale*.—A scale graduated in 0.02 in.

### Test Specimens

30. Three specimens approximately 10 in. in length shall be cut from the sample. The length of each specimen shall be measured within plus or minus 0.2 per cent.

### Procedure

31. The specimens shall be placed horizontally on rough paper toweling in an orderly fashion and in such a way that each makes contact only with the surface of the paper. The specimens on rough paper toweling shall be placed in the oven, in which the temperature shall be maintained at  $100 \pm 2$  C. for 2 hr. After the required time interval, the specimens shall be removed from the oven and kept at room temperature for 30 min. Each specimen shall be measured again, and the length recorded.

### Calculation

32. The percentage shrinkage shall be calculated as follows:

$$\text{Shrinkage, per cent} = \frac{L_1 - L_2}{L_1} \times 100$$

where:

$L_1$  = original length, and

$L_2$  = final length.

### Report

33. The report shall include the following:

- (a) Sample size from which the specimens were taken, and
- (b) Average percentage shrinkage.

### RESISTANCE TO PENETRATION AT ELEVATED TEMPERATURE

#### Apparatus

34. The apparatus shall consist of the following:

(a) *Penetration Tester*.—A penetration tester as shown in Fig. 1 is recommended. The component parts of the penetration tester are:

A stainless steel plate 4 by  $1\frac{1}{4}$  by  $\frac{1}{8}$  in.,

A  $\frac{1}{8}$ -in. magnetized steel rod, recessed at one end to hold a  $\frac{1}{16}$ -in. ball bearing against the test specimen or stainless steel plate,

the penetration tester and raising the temperature of the steel plate at a rate of 1 C. per 2 min.

(c) *Temperature Measuring Device.*—

A device for measuring the temperature

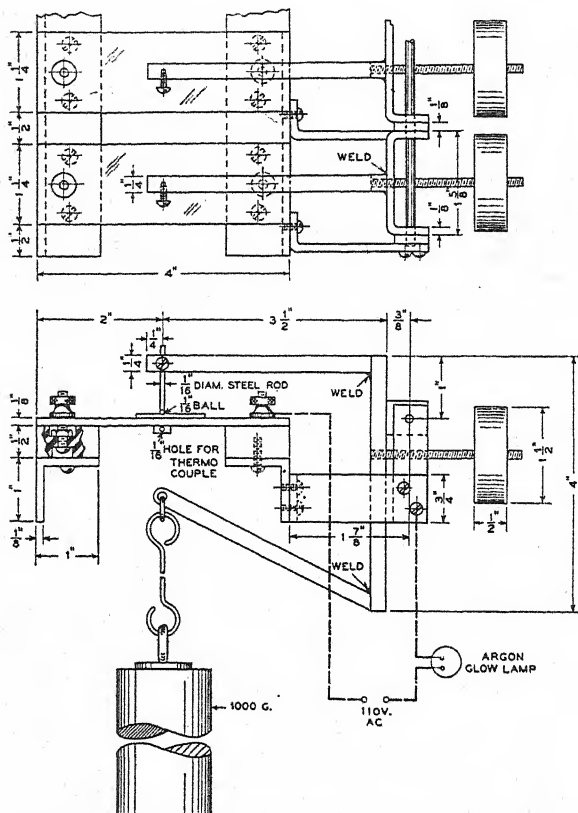


FIG. 1.—Penetration Tester for Determining Resistance to Penetration at Elevated Temperatures

A weight system capable of exerting a force of 1000 g. on the magnetized steel rod, including a counterbalance with a rider capable of being adjusted to neutralize the pressure of the ball bearing against the steel plate at no load,

A light C-clamp, containing the steel rod, counterbalance, and weight, mounted on a bearing capable of giving the unit the necessary freedom of rotation, and

An electrical circuit with a 110-v. a-c. supply and containing a 110-v. glow lamp.

(b) *Oven.*—An oven capable of holding

of the steel plate immediately below the point of contact of the ball bearing. A thermocouple is suggested for this application.

### Test Specimens

35. Five 1-in. specimens shall be cut from the sample and prepared for test by cutting the tubing open on one side along a longitudinal axis.

### Procedure

36. With no load on the rod, each specimen shall be inserted between the steel



ball and the steel plate, with the outside surface of the tubing facing the plate. The electric circuit shall be connected in such a way that when the steel ball comes in contact with the plate (when the specimen fails), the lamp outside the oven shall light. The compression load of 1000 g. shall then be applied to the specimen in the oven at room temperature (Note). The temperature of the steel plate shall be uniformly raised at the rate of 1 C. per 2 min. until failure of specimen is indicated by current flowing through the circuit and lighting the glow lamp outside the oven.

**NOTE.**—To facilitate testing, the initial starting temperature may be taken at 40 C. instead of room temperature. For convenience, five penetration testers may be constructed to test simultaneously the required number of specimens.

### Report

37. The report shall include the following:

- (1) Average wall thickness of the specimens,
- (2) Maximum and minimum temperatures at which the specimens failed, and
- (3) Average temperature of failure of the five specimens.

### INSULATION RESISTANCE TEST

38. The insulation resistance shall be determined (Note) in accordance with the Standard Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470),<sup>3</sup> except as specified in Sections 39 to 43.

**NOTE.**—Insulation resistance is not usually determined for tubing sizes above No. 0, but if necessary, larger sizes of tubing can be tested in accordance with the above method, provided the tubing is not kinked in the process of forming the U-shape.

### Apparatus

39. Besides the apparatus prescribed for the insulation resistance tests in Sections 20 to 23 of A.S.T.M. Methods D 470, additional apparatus shall con-

sist of a container for water, large enough to accommodate a specimen of tubing bent into a U-shape, and a support for the two branches of the U-shaped tubing specimen.

### Test Specimens

40. A test specimen of sufficient length to obtain a satisfactory reading on the galvanometer shall be cut from the sample. The minimum specimen length shall be 2 ft.

### Procedure

41. The specimen shall be shaped in a U-form and immersed in water at  $25 \pm 2$  C., containing 1 per cent sodium chloride. Not less than 1 ft. of the specimen shall be submerged. To avoid surface leakage the ends of the specimen shall protrude 6 in. above, and approximately perpendicular to, the surface of the water. The specimen shall be filled immediately with the same salt water, and, as a precaution against air bubbles and discontinuity of the water phase, a fine wire shall be inserted throughout the length of the specimen. The salt water in the tubing shall serve as an inner electrode, and its surface shall be above the surface of the salt water in the container. The salt water in the container shall serve as an outer electrode. The inner and outer electrodes shall be connected to the circuit. The specimen shall remain in the water for 24 hr. At the end of this time the insulation resistance shall be determined.

### Calculation

42. (a) In making insulation resistance calculations the length of tubing specimen submerged shall be used.

(b) When it is found necessary, the volume resistivity shall be calculated as follows:

$$R = 0.3663 \times \frac{\rho}{L} \times \log_{10} \frac{D}{d}$$



where:

$R$  = actual megohm reading,

$\varphi$  = volume resistivity in megohm centimeters,

$L$  = length of specimen in centimeters,

$D$  = outside diameter, and

$d$  = inside diameter.

## Report

43. The report shall include the following:

(1) Sample size from which the specimen was taken, and

(2) Insulation resistance of the tubing specimens in megohms per foot at 25 C. after 24 hr. immersion in salt water.

## DIELECTRIC STRENGTH TEST

44. The dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> except as modified in Sections 45 to 48.

## Apparatus

45. (a) *For Tubing Sizes No. 20 to  $\frac{1}{2}$  in., Inclusive, in Inside Diameter.*—Straight metal rods shall be used as inner electrodes. The nearest A.W.G. size of rod shall be selected that will fit tightly without stretching the tubing as it is being slipped onto the wire. Strips of metal foil shall be used as the outer electrodes. The strips shall be 1 in. in width and not more than 0.0005 in. in thickness.

(b) *For Tubing  $\frac{3}{8}$  to 2 in., Inclusive, in Inside Diameter.*—The test electrodes shall consist of opposing cylindrical rods  $\frac{1}{4}$  in. in diameter with edges rounded to a radius of  $\frac{1}{8}$  in. The upper movable electrode shall weigh  $0.1 \pm 0.006$  lb.

## Test Specimen

46. A test specimen long enough to obtain five breakdown voltages shall be cut from the sample.

## Procedure

47. (a) *For Tubing Size No. 20 to  $\frac{1}{2}$  in., Inclusive, in Inside Diameter.*—A length of tubing long enough to obtain one breakdown voltage shall be cut from the specimen and placed on the inner electrode. Part of the inner electrode shall be left exposed to make electrical connection. The tubing shall be long enough to accommodate an outer electrode, with enough tubing surface on either side of the outer electrode to prevent flashover. The outer electrode, consisting of a strip of metal foil, shall be tightly wrapped around the middle of the specimen. The first turn of the foil applied to the tubing shall be wiped tightly against it. Two more turns of the foil shall then be wound over the first turn. Allow a free end of  $\frac{1}{2}$  in. to which the electrical connection shall be made.

(b) *For Tubing  $\frac{3}{8}$  to 2 in., Inclusive, in Inside Diameter.*—The specimen shall be cut on one side along a longitudinal axis, flattened, and placed between the  $\frac{1}{4}$ -in. electrodes. The specimen shall be of sufficient area around the electrodes to prevent flashover.

(c) The dielectric strength test shall be made in transformer oil, free from foreign matter, and shall be determined by the short-time test. Voltage shall be applied between electrodes, and shall be increased at the rate of 0.5 kv. per sec.

(d) Five breakdown voltages shall be obtained on each specimen tested.

## Report

48. The report shall include the following:

(1) Sample size from which the specimen was taken,

(2) Total volts at each puncture,

(3) Maximum and minimum puncturing voltages, and

(4) Average dielectric strength.

## WET DIELECTRIC STRENGTH TEST

49. Wet dielectric strength shall be determined in accordance with Sections 43 to 47, except that a tubing sample of sufficient length shall be placed in tap water, with both ends protruding 1 in.

above the surface, at room temperature for a period of 24 hr. The sample shall be wiped or blotted to remove free water from the surface. The required specimen shall be immediately prepared and tested.

# *Tentative Methods of*

## TESTING VULCANIZED FIBRE USED FOR ELECTRICAL INSULATION<sup>1</sup>



A.S.T.M. Designation: D 619 - 46 T

ISSUED, 1941; REVISED, 1942, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover the procedures for testing vulcanized fibre sheets, tubes, and rods of such grades as can be used for electrical insulation.

### Definitions

2. (a) *Vulcanized Fibre*.—A material made by combining layers of chemically gelled paper, the gelling chemical being subsequently removed by leaching, resulting in a dense material of partially regenerated cellulose in which the fibre structure is retained.

NOTE.—Vulcanized fibre does not contain vulcanized rubber or sulfur as the name might imply.

(b) In referring to the cutting of the specimens and the application of the load, the following definitions apply:

*Flatwise*.—Load applied to the flat side of the original sheet.

*Edgewise*.—Load applied to the edge of the original sheet.

*Lengthwise*.—In the direction of the length of the sheet.

*Crosswise*.—In the direction at right angles to the length of the sheet.

NOTE.—When the sheet has the same length and width, one dimension shall arbitrarily be designated as the length, and the other as the width.

(c) In the case of thin material, the following definitions apply:

*Machine Direction*.—The lengthwise direction in which the paper is formed and travels on the paper machine wire or cylinder.

*Cross Direction*.—The direction at right angles to the machine direction.

NOTE.—These definitions conform to the practice of the paper industry.

### Conditioning

3. Vulcanized fibre shall be conditioned for physical tests in accordance with the Tentative Methods of Con-

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revision accepted by the Society through the Administrative Committee on Standards, November 23, 1946.

ditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618),<sup>3</sup> using the Standard Procedure for conditioning material 0.045 in. and under in thickness, and Functional Procedure A for conditioning material over 0.045 in. in thickness. In case of dispute in tests of materials over 0.045 in. in thickness, the material shall be exposed for 48 hr. at Standard Laboratory Atmosphere prior to conditioning by Functional Procedure A.

NOTE 1.—Conditioning of specimens may be undertaken (1) for the purpose of bringing the material into equilibrium with normal or average room conditions of 25 C. (77 F.) and 40 per cent relative humidity, (2) simply to obtain reproducible results regardless of the previous history of exposure, or (3) to subject the material to abnormal conditions of temperature or humidity in order to predict its service behavior.

The conditioning by Functional Procedure A prescribed in Methods D 618 to obtain reproducible results may give physical values somewhat higher or somewhat lower than the values at equilibrium under normal conditions, depending upon the test. This procedure for conditioning is used because of the relatively short time required. To bring the material to an equilibrium condition in a controlled humidity would require a long period of time which might extend over many months, for example, for thicknesses over 1 in. The exact length of time would depend upon such factors as thickness, grade, and previous history of the specimens, and it would be too long for ordinary commercial test purposes. It is probable that the time of exposure for some very thin sizes of material conditioned in accordance with the Standard Procedure may be reduced but sufficient data on the various thicknesses are not yet available to permit a decision to be made.

NOTE 2.—Conditioning of specimens for electrical tests is also necessary to obtain consistent results. The procedure for such conditioning is under consideration by Committee D-9. In order to secure comparable results, specimens should be conditioned at the same temperature and humidity.

## METHODS APPLICABLE TO SHEETS, TUBES, AND RODS

### Water Absorption

4. Rate of water absorption shall be determined in accordance with the Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570) of the American Society for Testing Materials.<sup>3</sup> The specimens shall be dried in an oven for 1 hr. at 105 to 110 C. prior to the immersion in water.

### Ash

5. Ash shall be determined in accordance with the procedure described in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials.<sup>3</sup>

### Silica

6. (a) *Test Specimen*.—The test specimen shall consist of approximately 3 g. of finely divided particles, such as millings or filings, of the vulcanized fibre.

(b) *Procedure*.—The test specimen shall be dried for 1 hr. at 105 to 110 C. The dried specimen shall be transferred to a crucible and slowly ignited to constant weight. The ash shall be wet with distilled water and transferred to a heat-resistant glass beaker. About 75 ml. of HCl (sp. gr. 1.19) shall be added and the beaker covered with a watch glass. The contents of the beaker shall be evaporated to dryness. To the residue shall be slowly added 10 ml. of HCl (sp. gr. 1.19) followed by 75 ml. of distilled water. The mixture shall be filtered through ashless filter paper and washed with cold water, then with warm water until the filtrate is free from chlorides. The filter paper shall be ignited to constant weight in a weighed platinum crucible. Then, 4 ml. of HF

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(48 to 60 per cent) shall be added and heat applied until all white fumes are driven off. The crucible shall be cooled and weighed. The difference between this weight and the previous one indicates silicon present as silica. The results shall be expressed as a percentage of the weight of the dried specimen.

### Volatile Matter

7. (a) *Purpose*.—This method is intended for the rapid determination, with reasonable accuracy, of the amount of moisture and other volatile matter in vulcanized fibre of all grades and thicknesses.

(b) *Test Specimen*.—The test specimen shall consist of the minimum number of pieces of fibre required to give a total weight of at least 1 g., and prepared as follows:

(1) *Sheets*.—For sheets less than  $\frac{1}{16}$  in. in thickness, the pieces for the test specimen shall be 3 by 1 in. by the thickness of the sheet; for sheets  $\frac{1}{16}$  in. and over in thickness, the pieces for the test specimen shall be 3 by  $\frac{1}{8}$  in. by the thickness of the sheet. The pieces shall be band-sawed or sheared from the sample so as to produce smooth edges free of cracks. The sawed faces shall be sanded or filed to remove any protruding sections that might be broken off during the test. The sample shall be sawed slowly so that the fibre is not heated appreciably. The thickness of the specimens shall be measured to the nearest 0.001 in. in the direction perpendicular to the natural faces of the original sample.

(2) *Tubes*.—For tubes less than  $\frac{1}{16}$  in. in wall thickness, the pieces for the test specimen shall be 1-in. lengths. For tubes  $\frac{1}{16}$  in. and over in wall thickness, the pieces for the test specimen shall be  $\frac{1}{8}$ -in. lengths slowly cut with a band saw.

(3) *Rods*.—For rods less than  $\frac{3}{16}$  in. in diameter, the pieces for the test specimen shall consist of continuous

lengths. For rods  $\frac{3}{16}$  in. and over in diameter, the pieces for the test specimen shall be  $\frac{1}{8}$ -in. lengths slowly cut with a band saw.

(c) *Procedure*.—Three test specimens, each consisting of one or more pieces of fibre as required, shall be tested individually. Each specimen shall be weighed to the nearest 1 mg., and placed in a mechanical convection oven maintained at a temperature of  $135 \pm 2$  C. and heated for the period prescribed in the following table. Specimens less than  $\frac{1}{8}$  in. in thickness shall be weighed (but not heated) in a weighing bottle.

	Size	Heating Period, hr.
Sheets.	Under $\frac{1}{16}$ in. in thickness.....	2
	$\frac{1}{16}$ and over in thickness.....	4
Tubes.	Under $\frac{1}{16}$ in. in wall thickness. . .	2
	$\frac{1}{16}$ in. and over in wall thickness	4
Rods.	Under $\frac{3}{16}$ in. in diameter.....	2
	$\frac{3}{16}$ in. and over in diameter ...	4

The specimens shall be removed from the oven, cooled in a desiccator, and weighed to the nearest 1 mg. The difference between the original weight and the final weight of the specimens shall be considered as the volatile matter content.

(d) *Calculation*.—The percentage of volatile matter content of the specimen shall be calculated as follows:

$$\text{Volatile matter, per cent} = \frac{\text{original wt. of specimen} - \text{final wt. of specimen}}{\text{final wt. of specimen}} \times 100$$

(e) *Report*.—The report shall include the following:

- (1) Color and dimensions of the specimen, and
- (2) Percentage of volatile matter.

### Zinc Chloride

8. (a) *Test Specimen*.—The test specimen shall consist of approximately 3 g. of finely divided particles, such as

millings or filings, of the vulcanized fibre.

(b) *Procedure*.—The test specimen shall be dried for 1 hr. at 105 to 110 C. The dried specimen shall be placed in a Soxhlet, Wiley-Richardson, or similar type of extractor and extracted with distilled water for 3 hr. The rate of extraction with the Wiley-Richardson size of extractor shall be sufficient to cause the tube containing the specimen to be filled by condensation and emptied by the siphon at least six times per hour. When the Soxhlet or similar size extractor is used, the tube shall be filled and emptied at a rate of at least three cycles per hour. After this extract has cooled, 0.5 ml. of potassium chromate solution (10 per cent) shall be added and the extract titrated with standard silver nitrate solution. The end point is indicated by the appearance of a permanent red color. A blank test shall be made on an equal volume of distilled water and potassium chromate solution and the result shall be subtracted from that of the specimen. The net result shall be calculated in terms of zinc chloride.

#### METHODS APPLICABLE TO SHEETS

NOTE.—In addition to the methods of testing sheets covered in the following Sections 9 to 20, inclusive, the methods described in Sections 4 to 8 are also applicable to sheet materials.

#### Tensile Strength

9. (a) For sheets 0.030 in. and under in thickness, the tensile strength shall be determined in accordance with the procedure described in the Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (A.S.T.M. Designation: D 202) of the American Society for Testing Materials.<sup>3</sup>

(b) For sheets over 0.030 in. in thickness, the tensile strength shall be determined in accordance with the procedure described in Standard Methods D 229.

#### Tearing Strength

10. Tearing strength of sheets 0.030 in. and under in thickness shall be determined in accordance with the procedure described in the Standard Method of Test for Internal Tearing Resistance of Paper (A.S.T.M. Designation: D 689).<sup>3</sup>

#### Compressive Strength

11. Compressive strength shall be determined in accordance with the procedure described in Standard Methods D 229.

#### Flexural Strength

12. Flexural strength of sheets  $\frac{1}{16}$  in. and over in thickness shall be determined in accordance with the procedure described in Standard Methods D 229.

#### Bursting Strength

13. Bursting strength of sheets under 0.045 in. in thickness shall be determined in accordance with the procedure described in Tentative Methods D 202.

#### Resistance to Impact

14. Impact strength shall be determined in accordance with the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256) of the American Society for Testing Materials.<sup>3</sup>

#### Density

15. (a) For sheets 0.020 in. and under in thickness, the specimen after conditioning shall be weighed on a chemical balance having a sensitivity of 1 mg. The thickness shall then be measured and the apparent density calculated from the thickness and weight as follows:

$$\text{Apparent density,} \\ \text{g. per cu. cm.} = \frac{\text{wt. in g.}}{\text{vol. in cu. in.}} \times 0.061$$

(b) For sheets over 0.020 in. in thickness, the density shall be determined with any suitable weight difference apparatus for making readings in water and air.

### Dielectric Strength

16. Dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials,<sup>3</sup> with the exceptions described in the procedure for dielectric strength in Standard Methods D 229.

### Rockwell Hardness

17. Rockwell hardness shall be determined in accordance with the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials,<sup>4</sup> with the additional provisions covered by the procedure for Rockwell hardness described in Standard Methods D 229. The R scale shall be used in all tests and the letter "R" shall precede all Rockwell hardness numbers.

### Thickness

18. (a) *On Test Specimens*.—All thickness measurements of test specimens shall be made using a machinist's micrometer in the manner outlined under method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

(b) *On Large Sheets*.—In the factory, thickness measurements of large sheets of material may be made by using a gage mounted on a yoke of sufficient

size and rigidity to permit accurate measurements of the thickness at the center of the sheet.

### Machine and Cross Directions

19. (a) *Test Specimen*.—A rectangular test specimen, approximately 2-in. square for sheets under  $\frac{1}{32}$  in. in thickness, and 1-in. square for sheets  $\frac{1}{32}$  in. and over in thickness shall be used.

(b) *Procedure*.—The specimen shall be measured in two directions at right angles to each other. The 2-in. square specimen shall be measured with a steel rule graduated in  $\frac{1}{16}$ -in. divisions, and the 1-in. square specimen shall be measured with a micrometer caliper. The specimens shall be immersed in water until swelling has occurred, removed, and remeasured immediately at the same locations that were measured before. The dimension which is found to show the greatest percentage increase during the immersion shall be considered the cross direction.

### Flammability

20. Flammability shall be determined in accordance with the procedure described in Standard Methods D 229.

### METHODS APPLICABLE TO TUBES

NOTE.—In addition to the methods of testing tubes covered in the following Sections 21 to 25, inclusive, the methods described in Sections 4 to 8 are also applicable to tubes.

### Tensile Strength

21. Tensile strength shall be determined in accordance with the procedure described in the Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 348) of the American Society for Testing Materials.<sup>3</sup>

### Compressive Strength

22. Compressive strength shall be determined in accordance with the pro-

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Parts I-A and I-B.

cedure described in Standard Methods D 348.

#### Density

23. Density shall be determined in accordance with the procedure described in Standard Methods D 348.

#### Dielectric Strength

24. Dielectric strength shall be determined in accordance with Standard Methods D 149, with the exceptions described in the procedure for dielectric strength in Standard Methods D 348.

#### Dimensional Measurements

25. Dimensional measurements of tubes shall be made in accordance with the Standard Methods of Measuring Dimensions of Rigid Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 668) of the American Society for Testing Materials.<sup>3</sup>

#### METHODS APPLICABLE TO RODS

NOTE.—In addition to the methods of testing rods covered in Sections 27 to 30, inclusive, the methods described in Sections 4 to 8 are also applicable to rods.

#### Tensile Strength

27. Tensile strength shall be deter-

mined in accordance with the procedure described in the Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (A.S.T.M. Designation: D 349) of the American Society for Testing Materials.<sup>3</sup>

#### Compressive Strength

28. Compressive strength shall be determined in accordance with the procedure described in Standard Methods D 349.

#### Flexural Strength

29. Flexural strength shall be determined in accordance with the procedure described in Standard Methods D 349.

#### Density

30. Density shall be determined in accordance with the procedure described in Standard Methods D 349.

#### Dimensional Measurements

31. Dimensional measurements of rods shall be made in accordance with the Tentative Methods of Measuring Dimensions of Rigid Rods Used in Electrical Insulation (A.S.T.M. Designation: D 741) of the American Society for Testing Materials.<sup>3</sup>



# Tentative Methods of TESTING ELECTRICAL INSULATING OILS<sup>1</sup>



A.S.T.M. Designation: D 117 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods of testing electrical insulating oils apply to mineral oil of petroleum origin for use in cables, transformers, oil circuit breakers, and other electrical apparatus as an insulating or cooling medium, or both. The test methods included are listed in Table I.

NOTE.—For oils having a Saybolt Universal viscosity over 150 sec. at 100 F. (37.8 C.), modifications of certain test procedures are necessary. These modifications are under study and development by Committee D-9. A notation appears in each test procedure where such modification is necessary.

## ACID AND BASE NUMBERS

### Definitions

2. (a) Properly refined insulating oils are free from mineral acids and alkalis. On the other hand, practically all oils contain natural organic substances which are either weak acids or which, upon oxidation, form compounds acidic in nature. The proportion of these acidic substances present in an oil is commonly referred to as its "organic acidity" to distinguish them from mineral (strong) acidity. "Organic acidity" is represented by the difference between total acid number and strong acid number. Additive-type oils may have an initial acid or alkaline reaction, distinctive of type of additive incorporated.

(b) *Total Acid Number*.—Total acid number is the number of milligrams of potassium hydroxide required to react with 1 g. of oil when tested as prescribed.

(c) *Strong Acid Number*.—Strong acid number is the number of milligrams

TABLE I.

Test	Definitions, Significance, and Procedure for Test Described in These Methods in Sections No.	A.S.T.M. Designation of Test Method
Acid and Base Numbers...	2 to 4	D 663, D 664
Color.....	5 to 7	D 155
Dielectric Strength.....	8 to 10	D 877
Flash Point.....	11 to 13	D 92
Free and Corrosive Sulfur.....	14 to 16	D 117 - 43
Inorganic Chlorides and Sulfates in Insulating Oils.....	17 to 19	D 878
Power Factor.....	20 to 22	D 150
Pour Point.....	23 to 25	D 97
Sampling.....	26 to 28	D 117 - 43
Saponification Number.....	29 to 31	D 94
Sludge.....	32 to 34	D 670
Specific Gravity.....	35 to 37	D 287, D 206
Steam Emulsion.....	38 to 40	D 157
Viscosity.....	41 to 43	D 88, D 445, D 446
Resistivity.....	44 to 46	D 257

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Administrative Committee on Standards, November 23, 1946.

These methods are in effect a tentative revision of, and, when adopted, are intended to replace the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation D 117 - 43), see p. 65.

of potassium hydroxide required to react with the strong acid content of 1 g. of oil under prescribed conditions.

(d) *Strong Base Number*.—Strong base number is the number of milligrams of potassium hydroxide equivalent to the acid required to react with 1 g. of oil when tested according to the method.

### Significance

3. (a) In the inspection of unused oils, strong acid and strong base number values are important as a check against the presence of mineral acidity or alkalinity. Appreciable quantities of organic acidity may be detected by the difference between total acid number and strong acid number.

(b) In used insulating oils total acid number may be pertinent, if compared to the value for the unused product, to detect contamination by substances with which the oil has been in contact, to reveal tendency toward increase in acidity due to inherent chemical change, or to indicate chemical changes in additive. An increase in total acid number may be used as a general guide for determining when a particularly well-known oil should be replaced by fresh material to prevent further decomposition and consequent sludging, provided suitable rejection limits have been established and other tests confirm the need for a change.

### Procedure

4. Acid and base numbers shall be determined in accordance with the Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 663)<sup>3</sup>. Acid and base numbers may also be determined in accordance with the Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration (A.S.T.M. Designation: D

664),<sup>3</sup> if it is desired to use an electro-metric titration method.

### COLOR

#### Definition

5. The color of an insulating oil is determined by means of transmitted light, and is expressed by a numerical value based on comparison with a series of color standards.

#### Significance

6. (a) The chief significance of color as applied to an insulating oil lies in the fact that it is the generally accepted index of the degree of refinement for unused oils and an approximate measure of deterioration of products in service. Increase in color number may be indicative of contamination.

(b) While there may be some connection between the color of an insulating oil and its degree of refinement, there has never been established a relationship between this characteristic and physical, chemical, or electrical stability. Therefore, color by itself may be considered of little inherent value.

#### Procedure

7. Color shall be determined in accordance with the Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155).<sup>3</sup>

### DIELECTRIC STRENGTH

#### Definition

8. The dielectric strength of an insulating material may be defined as the minimum voltage gradient at which electrical failure or breakdown occurs under prescribed conditions.

#### Significance

9. The dielectric strength of an insulating oil is of importance as a measure

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

of its ability to withstand electric stress without failure. It may also serve to indicate the presence of contaminating agents, such as water, dirt, or conducting particles in the oil, one or more of which may be present simultaneously when low dielectric strength values are found by test. However, a high dielectric strength is not a certain indication of the absence of all contaminants.

### Procedure

10. Dielectric strength shall be determined in accordance with the Tentative Method of Test for Dielectric Strength of Insulating Oils (A.S.T.M. Designation: D 877).<sup>4</sup>

### FLASH POINT

#### Definition

11. The flash point of a material which emits combustible vapor may be defined as the temperature to which the material must be heated in order to give off sufficient vapor to form a flammable mixture with air.

#### Significance

12. The flash point of a mineral insulating oil reveals the limit to which the material may be heated under the specified test conditions before the vapors emitted became a fire hazard. An unusually low flash point for a given product indicates contamination.

#### Procedure

13. Flash point shall be determined in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>3</sup>

### FREE AND CORROSIVE SULFUR

#### Definition

14. (See Footnote 5.)

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>5</sup> Work is in progress on the development of statements on definition and significance of this test and they will be added in future revisions as rapidly as completed by the committee.

### Significance

15. (See Footnote 5.)

### Procedure<sup>6</sup>

16. The recommended procedure for conducting the test for free and corrosive sulfur is that described in the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117-43),<sup>4</sup> except that the copper strip shall be immersed in the oil at  $100 \pm 2^\circ\text{C}$ ., for a period of 5 hr., after which time the glass tube shall be withdrawn from the bath and the copper strip examined.

### INORGANIC CHLORIDES AND SULFATES IN INSULATING OILS

#### Definition

17. (See Footnote 5.)

#### Significance

18. (See Footnote 5.)

#### Procedure

19. The presence of inorganic salts (chlorides and sulfates) shall be determined in accordance with the Tentative Method of Test for Inorganic Chlorides and Sulfates in Insulating Oils (A.S.T.M. Designation: D 878).<sup>4</sup>

### POWER FACTOR

#### Definition

20. (See Footnote 5.)

#### Significance

21. (See Footnote 5.)

#### Procedure<sup>7</sup>

22. Power factor shall be determined in accordance with the Tentative Method of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>4</sup>

<sup>6</sup> A new method for the detection of free and corrosive sulfur is in the course of preparation and when completed will be issued under a separate A.S.T.M. designation.

<sup>7</sup> A new method of test for power factor is in course of preparation and when completed will be issued under a separate A.S.T.M. designation.

The cell to be used for testing the oil shall be suitable for measuring power factors ranging between values of 0.0001 and 0.25.

#### POUR POINT

##### Definition

23. Insulating oils become more or less plastic solids when sufficiently cooled, due either to partial separation of wax or to congealing of the hydrocarbons or derivatives composing the products. The temperature at which the liquid just flows under the prescribed conditions is known as the pour point.

##### Significance

24. (a) The pour point of an insulating oil gives an indication of the temperature below which it may not be possible to pour or remove the oil from its container.

(b) In connection with oil for oil-filled cables, the pour point may be useful to indicate the point at which no free movement will take place in the cable, or to indicate the temperature at which partial separation of wax may begin.

(c) The pour point of a transformer oil is important as an index of the lowest temperature to which the material may be cooled without seriously limiting the degree of circulation of the oil in the transformer casing.

##### Procedure

25. Pour point shall be determined in accordance with the Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97).<sup>3</sup>

#### SAMPLING

##### Definition

26. Sampling is the obtaining of an adequate portion which is representative of an entire quantity or of a particular cross-section of material. In some cases the detection of contaminants which are not ordinarily dispersed uniformly in an oil, such as water or rust, necessitates

taking samples at specific locations where the contaminants are likely to be found.

##### Significance

27. Accurate sampling, whether of the complete contents or only parts thereof, is extremely important from the standpoint of evaluation of the quality of the product sampled. Obviously, examination of a sample that, because of careless sampling procedure or contamination in the sampling equipment, is not directly representative, leads to erroneous conclusions concerning quality and, in addition, results in a loss of time, effort and expense involved in securing, transporting, and testing the sample.

##### Procedure<sup>3</sup>

28. Sampling shall be carried out in accordance with the procedure described in the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117-43).<sup>4</sup>

#### SAPONIFICATION NUMBER

##### Definition

29. The saponification number of an oil is the number of milligrams of potassium hydroxide which is consumed by one gram of oil under the prescribed conditions.

##### Significance

30. The saponification number is the best index of the percentage of fat or fatty oil in a given product. In oils containing no fat or fatty oil, the test merely measures acidic compounds present which react with potassium hydroxide. Since the limit of error of the test (plus or minus 0.5) is greater than the amount of saponifiable material normally present in electrical insulating oils, the value and significance of saponification number for the evaluation of the quality of these products is questionable.

<sup>3</sup> A revised method of sampling insulating oils is in the course of preparation and when completed will be issued under a separate A.S.T.M. designation.

### Procedure

31. Saponification number shall be determined by the Standard Method of Test for Saponification Number of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 94).<sup>4</sup>

### SLUDGE

#### Definition

32. The sludge value for mineral transformer oil represents the percentage of insoluble matter formed when the oil is heated and oxidized under certain prescribed conditions.

#### Significance

33. (a) Sludge in transformer oil tends to deposit upon transformer parts and interfere with heat transfer, and to choke oil ducts and so hinder oil circulation and heat dissipation. Sludge also tends to increase water retentivity, lower dielectric strength, impair power factor, and increase the rate of further chemical reaction in the oil itself. An oil which, among other things, reveals a high degree of resistance to sludge formation is considered desirable for transformer use, although the oil possessing the lowest sludge test value is not necessarily the best. Additional physical, chemical and electrical factors, including the rate of corrosive acid formation, must be considered in the final decision as to the usability of the oil in transformers.

(b) Numerical correlation between results from these laboratory sludge tests and years of service and sludge accumulation in transformers has yet to be established.

#### Procedure

34. Sludge value shall be determined in accordance with the Tentative Methods of Test for Sludge Formation in Mineral Transformer Oil (A.S.T.M. Designation: D 670).<sup>4</sup>

### SPECIFIC GRAVITY

#### Definition

35. The specific gravity of an oil is, according to the general custom of this country, the ratio of the weights of equal volumes of oil and water, both weights being determined with the liquid at a temperature of 60 F. (15.56 C.) and both weights being corrected for the buoyant effect of air. This characteristic is expressed as "Specific Gravity 60/60 F." wherein the first 60 F. refers to the temperature of the oil and the second 60 F. refers to the temperature of the water.

#### Significance

36. Electrical insulating oils are usually sold on the basis of volume delivered at 60 F. Delivery is often made on the basis of net weight of product in drums, and the specific gravities are often measured at temperatures other than that of 60 F. The value of specific gravity at 60 F. must be known to compute the volume at 60 F. of the oil delivered.

Specific gravity is not significant as such in determining the quality or suitability of the insulating oil for a given application.

#### Procedure

37. Specific gravity shall be determined in accordance with the Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287).<sup>3</sup> Conversion of readings taken at temperatures other than 60 F. shall be made in accordance with either the abridged table appearing in A.S.T.M. Method D 287, or the Standard Abridged Volume Correction Table for Petroleum Oils (A.S.T.M. Designation: D 206),<sup>3</sup> or U. S. Bureau of Standards *Circular C 410*. The test report shall state which conversion table was used.

## STEAM EMULSION

### Definition

38. (a) When a mineral oil and water are mixed in any manner, they generally tend to separate more or less rapidly on standing. The presence of small amounts of certain other materials, particularly polar compounds, decreases the rate of separation and may prevent any separation whatever. This physical condition is known as an emulsion, and the phenomenon is known as emulsification.

(b) In the steam emulsion test, steam is passed into the oil under prescribed conditions and the time in seconds required for complete separation of the oil and the condensed steam is recorded as the Steam Emulsion Number (S.E. No. ....) of the oil.

### Significance

39. The A.S.T.M. test for steam emulsion was developed to predict the tendency of a mineral oil to resist emulsification in the event it should come in contact with steam or water during use. In the case of an insulating oil, high S. E. Number would reflect slow rate of separation from water and would indicate the presence of substances which might eventually depreciate the insulating properties of the oil. However, because insulating oils are not ordinarily subjected to conditions which develop emulsification, there is no uniformity of opinion as to the value and significance of the test.

### Procedure

40. Steam emulsion number shall be determined in accordance with the Standard Method of Test for Steam Emulsion of Lubricating Oils (A.S.T.M. Designation: D 157).<sup>3</sup>

### VISCOSITY

### Definition

41. The viscosity of a fluid is its

resistance to uniformly continuous flow, without turbulence, inertia, or other forces. The viscosity of insulating oil is usually measured by the time of flow of a given quantity of oil under controlled conditions.

### Significance

42. (a) The viscosity of the insulating oil used in solid type cables, at the temperature used for impregnation, is a factor in the rate of impregnation. The viscosity at operating temperatures is the principal factor in the mobility of the insulating oil under service conditions.

(b) The viscosity of a transformer oil at operating temperatures is a principal factor in the convection flow of the oil needed for heat dissipation.

### Procedure

43. Viscosity shall be determined in accordance with either the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88)<sup>3</sup> or the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445)<sup>3</sup> and, unless otherwise specified, at a temperature of 37.8 C. (100 F.). The conversion of viscosity values may be made in accordance with the Standard Method for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity (A.S.T.M. Designation: D 446).<sup>3</sup>

## RESISTIVITY

### Definition

44. (See Footnote 5.)

### Significance

45. (See Footnote 5.)

### Procedure

46. Resistivity shall be determined in accordance with the Standard Method of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257).<sup>4</sup>

# *Tentative Methods of* TESTING ASKARELS<sup>1</sup>



A.S.T.M. Designation: D 901 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods of testing apply to askarels, synthetic nonflammable type insulating liquids which, when decomposed by the electric arc, evolve only nonexplosive gaseous mixtures.

NOTE.—Various materials have been used successfully in taking, storing, and testing samples of askarels. Until experience has developed a strong preference, materials referred to in these methods will be understood to be approved but not specified.

## SAMPLING

### General

2. Samples from shipments of askarels shall be taken in such a manner that they will be representative of the shipment. Samples from askarels in service shall be taken in such a manner that they will represent the portion of liquid in poorest condition.

### Sampling Apparatus

3. (a) *General*.—Sampling apparatus shall be made of materials which will have no appreciable effect upon the test results. Approved materials are glass, stainless steel, or chromium-plated metal. Sampling apparatus shall be specially constructed for consistently withdrawing samples from various types of containers or electrical apparatus.

NOTE 1.—The sampler shown in Fig. 1 is so constructed that it may be used in taking either a top or a bottom sample. In either case the sample is released at the bottom of the sampler.

NOTE 2.—The sampler shown in Fig. 2 is of the cream dipper type. Liquid enters the dipper at the top and is released into the sample bottle by opening the valve at the bottom.

NOTE 3.—The sampler shown in Fig. 3 is of the vacuum type. It consists of a bottle fitted with a 2-hole cork stopper, glass tubing and a syringe bulb. Liquid is drawn through the glass tubing into the bottle by means of a partial vacuum produced by operation of the bulb.

(b) *For Drums*.—For sampling drums the apparatus shall be so designed that liquid will enter the top of the sampler cup when the cup is submerged approximately  $\frac{1}{4}$  in.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

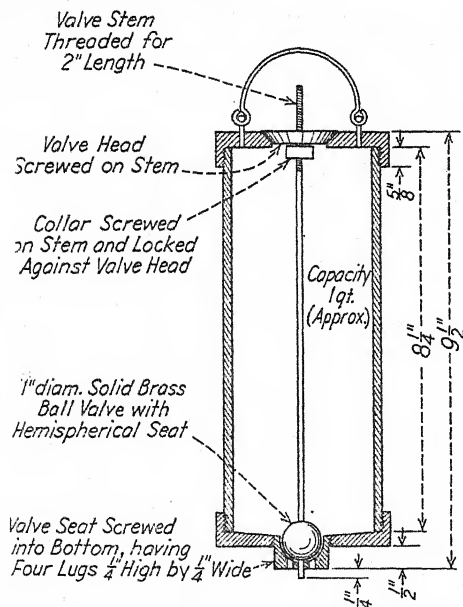
<sup>2</sup> Accepted by the Administrative Committee on Standards, November 23, 1946.

Prior to their acceptance as tentative, these methods were published as information from 1943 to 1946, being revised in 1945.

NOTE 4.—A convenient and simple device for sampling drums is of the cream dipper type shown in Fig. 2.

(c) *For Cans.*—For sampling cans of 5-gal. capacity or larger an apparatus of the dipper type, Fig. 2 may be used, or an apparatus of the vacuum type shown in Fig. 3 may be used.

(d) *For Tank Cars.*—For sampling tank cars the sampling apparatus may be of the type shown in Fig. 1. This is a modification of the type shown in Fig. 2 of the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117).<sup>3</sup>



NOTE.—Thief may be used for taking top or bottom sample from tank car. For bottom sample use as shown in sketch. For top sample screw valve head and collar about  $\frac{1}{4}$ -in. up on stem so that top valve remains fixed in open position, and submerge thief just below liquid level.

FIG. 1.—Sampler for Tank Cars.

## Sample Containers

4. (a) The following may be used as containers for the samples:

- (1) Tin cans,
- (2) Cork-stoppered or glass-stoppered dark-glass bottles, and

\* Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

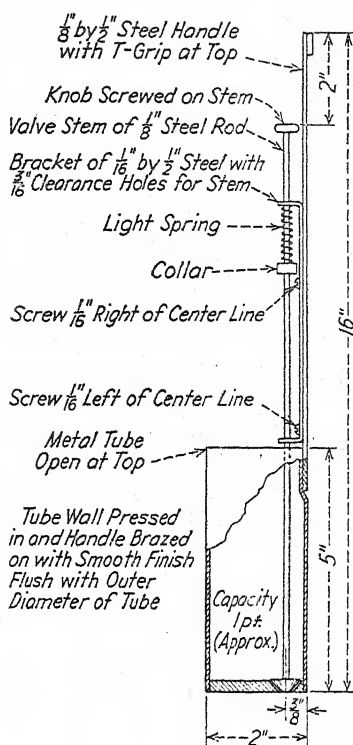
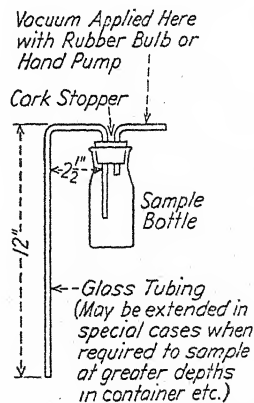


FIG. 2.—Sampler for Drums.



NOTE.—In the case of samples for extremely sensitive tests such as power factor there is some evidence to indicate that brass or copper thefts may affect the test results. The chemically clean glass thief is used to take samples for such tests from both 5-gal. cans and drums of new material. In special cases, it is used to sample apparatus in service.

FIG. 3.—Glass Sampler for Cans.



- (3) Cork-stoppered or glass-stoppered clear-glass bottles in light-proof cartons.

NOTE.—The dark-glass bottle has the advantage that it protects the sample from the effects of bright light. The clear-glass bottle offers the advantage that it may be examined to see that it is clean. It also allows visual inspection of the liquid before testing, permitting the detection of free water or solid impurities.

(b) The containers shall be clean and dry. Samples placed in glass bottles shall be kept in the dark if subsequent testing is to include color, neutralization value, power factor or resistivity. For this reason light-proof cartons with covers shall be provided for each bottle into which the bottle with its sample of liquid shall be placed immediately after sampling. If cork stoppers are used they shall be covered with tin or aluminum foil. If glass-stoppered bottles are used, precaution shall be taken to insure that perfectly fitting stoppers are secured. A tin can fitted with a screw cap protected with a cork disk faced with tin or aluminum will endure harder usage. Tin cans shall be of the type having seams soldered on the exterior surfaces.

#### Cleaning of Sampling Apparatus and Sample Containers

5. (a) *General*.—All sampling apparatus and sample containers shall be cleaned thoroughly before using, special care being taken that no lint or other fibrous material remains in or on them. Care shall be taken to insure the complete removal of all traces of soldering flux from new containers.

NOTE.—Alcohol is a good solvent for soldering flux.

(b) *Sampling Apparatus*.—Sampling apparatus, before using, shall be cleaned by rinsing with dry, lead-free gasoline, Stoddard solvent, or carbon tetrachlo-

ride, and dried in the hot cabinet of the type described in Section 6 (a). The best procedure is to rinse it after the sampling has been finished and then place it in the hot cabinet, as in this way it will be ready for use without further cleaning when the next samples are to be taken. The rinsing material shall be used but once and then discarded.

(c) *Sample Containers*.—Sample containers, before using, shall be rinsed with lead-free gasoline and dried. They shall then be washed with strong soapsuds, rinsed thoroughly with distilled water and then dried by passing a current of dry, clean, warm air through the containers, after which they shall be immediately placed in the hot cabinet described in Section 6 (a). A similar washing will be sufficient for re-use of the containers that have held new liquid but those which have contained used liquid shall be washed with both lead-free gasoline and benzol before being washed with soapsuds.

#### Storage of Sampling Apparatus and Sample Containers

6. (a) *General*.—After cleaning and until required, all sampling apparatus and sample containers shall be kept in a dry, dust free, hot cabinet in which an air temperature of approximately 60 C. shall be maintained.

(b) *Sampling Apparatus*.—Sampling apparatus shall be kept at all times in a vertical position in the hot cabinet in a rack having a suitable drainage receptacle at the base.

(c) *Sample Containers*.—Containers for samples, after being washed and dried as described in Section 5 (c), shall be kept unstoppered while in the hot cabinet and shall be sealed immediately on removal. During the preparation and subsequent handling of the container when introducing the sample, care shall be taken that the operator's fingers do not touch the lip of the container.

### Procedure for Sampling New Askarel

7. (a) It is recommended that 1 qt. of liquid be taken as a sample if complete physical and chemical tests are to be made and 1 pt. if only dielectric strength tests are to be made. Sample containers should not be completely filled, about 10 per cent of the volume being left for expansion.

(b) Samples shall not be taken from containers until the liquid is at least as warm as the surrounding air.

(c) Sample containers and sampling apparatus shall be rinsed with the liquid about to be sampled.

(d) Drums and cans shall be preferably sampled indoors and shall be kept undisturbed for at least 12 hr. before samples of liquid are taken.

(e) Drums of liquid to be sampled shall be lined up, preferably on their sides with their bungs up. The bungs shall be unsealed, removed and laid with the liquid contact sides up beside the bungholes. The dipper type sampler (Fig. 2) shall be lowered into the liquid until the lip of the cup is approximately  $\frac{1}{4}$  in. below the surface of the liquid. When the sample has been transferred to the sample container, both the drum and the sample container shall be closed at once.

(f) When sampling containers such as cans of 5-gal. capacity or other small containers with the dipper type sampler (Fig. 2), the sampler shall be manipulated as in sampling drums or barrels. When using the vacuum type sampler (Fig. 3) the length of stem below the stopper shall be so adjusted that air bubbles are entrained as the sample is being drawn. This insures inclusion of a portion of the "surface layer." When the sample has been taken, the liquid container and the sample container shall be closed at once.

(g) Tank cars of liquid shall be sampled by means of a sampler of the type

shown in Fig. 1. Both top and bottom samples shall be taken, as follows:

(1) In taking a top sample the upper valve head shall be so adjusted on the threaded shaft that it is held about  $\frac{1}{4}$  in. above the top of the sampler. The sampler shall be lowered into the liquid so that the top is approximately  $\frac{1}{4}$  in. below the liquid surface. The sample shall be transferred to the sample container by lifting the valve stem and so opening the lower valve.

(2) In taking a bottom sample the upper valve head shall be screwed down upon the collar in which position both valves remain closed until the stem is lifted. Displacement of air through the top valve largely prevents inflow of liquid through the top valve so that the sample is essentially a bottom sample. The sample container shall be stoppered immediately after it has been filled.

(h) When samples are being taken from different sources, precautions shall be taken to clean the sampler so as to prevent possible contamination of samples.

(i) Samples in either dark-glass or clear-glass bottles shall be stored in the dark.

### Procedure for Sampling Liquids in Service

8. (a) It is recommended that 1 qt. of liquid be taken as a sample if complete physical and chemical tests are to be made and 1 pt. if only dielectric strength tests are to be made. Sample containers should not be completely filled, about 10 per cent of the volume being left for expansion.

(b) The precautions outlined in Sections 5 and 6 regarding the cleaning and storage of sampling apparatus and sample containers shall be observed as far as practicable.

NOTE 1.—As a precaution against moisture absorption, unnecessary exposure of askarel to the atmosphere should be avoided.

(c) Sample containers and sampling apparatus shall be rinsed with the liquid about to be sampled.

(d) The sampling of askarel in service is complicated by the fact that samples for electrical tests must be taken at the top surface of the liquid while samples for sediment must be taken from the bottom. Many transformers are equipped with a special sampling valve located just below the surface of the liquid. This valve terminates in a bent tube, open at the end, inside the transformer. This tube may be rotated about the horizontal center line of the valve as an axis, by a control knob on the outside of the transformer. The tube connects to an outlet on the outside of the transformer, the opening normally being capped. When not in use this tube is turned so that it points vertically upward. In this position the open end is above the surface of the liquid.

To take a sample the outside cap shall be removed and the tube rotated to a horizontal position. With the tube held in this position, just enough liquid shall be drawn off to flush the tube. This shall not be drawn into the sampling container. After the flushing, the tube shall be returned to the vertical position and the sample container substituted for the receptacle used for flushing. With the sample container held under the outlet, the tube shall then be turned until liquid just begins to flow freely through the tube. The tube shall be held in this position until a sufficient sample has been withdrawn; it shall then be returned to the vertical position.

NOTE 2.—In humid weather it is advisable to attach a metal fitting (cleaned as specified in Section 5) to extend from the valve into the sample container to prevent contamination of the sample by atmospheric moisture.

(e) Apparatus not equipped with special sampling valves may be sampled

either by means of the dipper type sampler (Fig. 2) or the vacuum type sampler (Fig. 3). In using the vacuum type sampler, the end of the tube in the transformer shall be so adjusted that air bubbles are entrained as the sample is being drawn.

(f) Samples in either dark-glass or clear-glass bottles shall be stored in the dark.

## SPECIFIC GRAVITY

### Definition

9. The specific gravity of askarels is the ratio of the weight of a given volume of the material at a temperature of 60 F. (15.56 C.) to the weight of an equal volume of distilled water at the same temperature, both weights being corrected for the buoyancy of air.

### Apparatus

10. (a) *Hydrometer*.—A hydrometer of the conventional floating glass type, calibrated in terms of specific gravity shall be used. The range of the instrument shall be any suitable portion of the interval between 1.0000 and 2.0000, with the scale divided in 0.001 of a unit of specific gravity, and the scale divisions spaced not less than 0.75 mm. (0.03 in.) nor more than 1.5 mm. (0.06 in.) apart. The scale shall be so marked that it can be easily read, and the accuracy of the calibration of the hydrometer shall be such that the error at any point of the scale shall not exceed 0.0005.

(b) *Hydrometer Cylinder*.—The receptacle in which the liquid is tested shall be a cylinder made of clear glass. The inside diameter of the cylinder shall be at least 2.54 cm. (1.0 in.) greater than the outside diameter of the hydrometer and the height of the cylinder shall be such that the column of liquid under test exceeds by at least 2.54 cm. (1.0 in.) the submerged portion of the hydrometer after a state of equilibrium has been reached.

(c) *Thermometer*.—An A.S.T.M. ther-

mometer of the total immersion type and conforming to the requirements prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1),<sup>3</sup> shall be used in taking the temperature of the liquid under test.

(d) *Temperature-Control Apparatus.*—Suitable apparatus shall be provided for controlling the temperature of the liquid under test to  $60 \pm 1$  F ( $15.56 \pm 0.56$  C.). This may consist of a liquid bath, into which the hydrometer cylinder is placed, or a conditioning chamber in which temperature-controlled air is the regulating medium.

#### Procedure

11. (a) The measurement of specific gravity shall be made at a temperature of 60 F. (15.56 C.). The sample to be tested shall be poured into the clean hydrometer cylinder without splashing, so as to avoid, as much as possible, the formation of air bubbles. If air bubbles are formed, they shall be removed after they have collected on the surface by touching them with a piece of clean blotting or filter paper before the hydrometer is placed in the sample. The hydrometer and thermometer shall then be placed in the liquid and the cylinder placed in the conditioning medium and kept there until the required temperature of sample as indicated by the thermometer, is reached. An occasional gentle stirring will facilitate reaching the desired temperature. If a liquid bath is used, the apparatus should be set up in a location as free from air currents as possible. If an air conditioning chamber is used, any fans used for air circulation shall be shut off as soon as the test temperature is reached, so that disturbance from air currents will be minimized.

(b) When the liquid under test has reached the test temperature, the hydrometer shall be raised slightly and the stem carefully wiped clean, after which it shall be lowered slowly into the sample

to a level of two smallest scale divisions below that at which it will float, and shall then be released. When the hydrometer has come to rest, floating freely away from the walls of the cylinder, the specific gravity shall be read and recorded. In the case of transparent liquids, the hydrometer shall be read with the eye at the level of the surface of the liquid and at the point where the bottom of the meniscus of the liquid intersects the hydrometer scale. In the case of nontransparent liquids, the hydrometer shall be read at the highest point on the scale to which the liquid rises. This reading shall then be corrected by an amount equal to the height to which the liquid rises on the hydrometer scale above the main surface of the sample.

(c) The thermometer in the sample shall be read immediately after the specific gravity is determined.

#### Reproducibility of Results

12. (a) Duplicate determinations shall agree within 0.001 unit of specific gravity when the same hydrometer and thermometer are used in the tests.

(b) Separate laboratories using different hydrometers and thermometers should obtain duplicate results, agreeing within 0.002 unit of specific gravity.

#### COLOR

##### Preparation of Reference Standards

13. (a) Dissolve 1.245 g. of potassium chloroplatinate ( $K_2PtCl_6$ ) containing 0.5 g. of platinum, and 1 g. of crystallized cobaltous chloride ( $CoCl_2 \cdot 6H_2O$ ) containing about 0.25 g. of cobalt in water with 100 ml. of HCl (sp. gr. 1.18). Dilute to 1 liter with distilled water. This solution has a color of 500.

(b) To prepare reference standards having colors of 5, 10, 15, 20, 25, 30, 35, etc., dilute 0.5, 1.0, 1.5 ml., etc., of the solution prepared as described in Paragraph (a) with distilled water to

50 ml. in 50-ml. tall-form Nessler tubes. Protect the reference standards from evaporation when not in use.

(c) The unit of color is that color produced by 1 mg. of platinum per liter.

#### Procedure

14. (a) The color of the sample shall be observed by filling a matched Nessler tube to a height equal to that in the reference standard tubes with the liquid to be examined and comparing it with the reference standards. The determination shall be made by looking vertically downward through the tubes upon a white or mirrored surface placed at such an angle that light is reflected through the columns of liquids.

(b) This method is good only up to a color intensity of about 100. For reading higher values the sample should be diluted with water-white benzol and the color obtained by multiplying by the dilution ratio.

#### Recording Colors

15. Colors between 1 and 100 shall be recorded to the nearest 5. Colors between 101 and 300 shall be recorded to the nearest 10. Colors above 300 shall be recorded in accordance with the Tentative Method of Test for Color of Lubricating Oil and Petrolatum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155).<sup>4</sup>

#### VISCOSITY

##### Procedure

16. Viscosity shall be determined in accordance with either the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88)<sup>4</sup> or the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445)<sup>4</sup> and, unless otherwise specified, at a temperature of 37.8 C. (100 F.).

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

#### FIRE POINT

##### Procedure

17. Askarels as such have no fire point. As a test for appreciable contamination by flammable liquids the lack of a fire point shall be verified in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>4</sup> The verification temperature shall be 200 C. (392 F.).

#### POUR POINT

##### Procedure

18. Pour point shall be determined in accordance with the Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97).<sup>4</sup>

#### NEUTRALIZATION NUMBER

##### Procedure

19. Neutralization number shall be determined in accordance with the Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Color-Indicator Titration (A.S.T.M. Designation: D 663).<sup>4</sup> Neutralization number may also be determined in accordance with the Tentative Method of Test for Acid and Base Numbers of Petroleum Products by Electrometric Titration (A.S.T.M. Designation: D 664)<sup>4</sup> if it is desired to use an electrometric titration method.

#### FREE CHLORIDES

##### Nature of Test

20. This method applies to the quantitative determination of the free chlorides in askarels.

##### Extraction of Chlorides

21. (a) A 50-ml. sample of askarels shall be heated to 90 to 100 C. (194 to

212 F.), placed in a chemically clean 100-ml. separatory funnel and extracted with two portions of hot (90 to 95 C. (194 to 203 F.)) chloride-free distilled water. The total weight of water shall be equal to that of the 50 ml. of askarel tested. The first extraction shall be made using approximately two thirds of the water. The second extraction shall be made using the remaining one third of the water.

(b) Extraction shall be made by stoppering the funnel and shaking the askarel-water mixture vigorously for a minimum of 20 sec. After allowing to settle, the water extracts shall be separated into a clean centrifuge flask. The first and second extracts shall then be combined and mixed thoroughly.

(c) The combined water extracts shall be centrifuged until the water shows a negative Tyndall beam effect.

#### Apparatus

22. (a) *Burette*.—A 10-ml. burette graduated in subdivisions not greater than  $\frac{1}{10}$  ml.

(b) *Evaporating Dish*.—A clean white porcelain evaporating dish of at least 150-ml. capacity.

#### Reagents

23. (a) *Standard Sodium Chloride Solution*.—Dissolve 0.824 g. of pure fused sodium chloride in distilled water and make up to 1 liter. Dilute 100 ml. of this solution to 1 liter. One milliliter of this dilute solution contains 0.05 mg. of chloride radicle. Distilled water shall be chlorine free.

(b) *Standard Silver Nitrate Solution*.—Dissolve about 0.24 g. of silver nitrate crystals in 1 liter of distilled water. Titrate against the above standard sodium chloride and adjust silver nitrate solution so that 1 ml. will be exactly equivalent to 0.05 mg. of chloride radicle.

(c) *Potassium Chromate Indicator*.—

Dissolve 50 g. of neutral potassium chromate in a little distilled water. Add silver nitrate to produce a slight red precipitate. After a day or two, filter and dilute the filtrate to 1 liter with distilled water.

#### Procedure

24. Prepare a solution for the purpose of color reference by adding 1 ml. of chromate indicator to 50 ml. of distilled water in a clean evaporating dish, and titrate with the standard silver nitrate, adding the solution drop-by-drop while stirring constantly until a very slight reddish tinge, when compared with the reference standard, is produced that will persist for several minutes. The end point is very sharp if the test is performed in a darkened room provided with a yellow light.

NOTE.—In case the chloride content is less than 5 ppm. a large amount of askarel should be treated to give 100 ml. of water extract for test and the proper factor applied.

#### Report

25. The chloride content shall be reported in parts per million which is numerically equal to the milliliters of silver nitrate used in the test.

#### ARC-FORMED GASES

##### Apparatus

26. The apparatus shall consist of the following:

(a) *Glass Container*.—A glass container of not less than 2-oz. capacity, fitted with suitable electrodes and outlet for gases.

(b) *Glass Tube*.—Glass tube filled with lightly-packed glass wool for removal of carbon particles from gases of decomposition.

(c) *Graduated Container*.—Graduated container for collection of gases by displacement of a suitable liquid.

(d) *Gas Apparatus*.—An Orsat or Burrell apparatus for analysis of the gases.



### Procedure

27. (a) Any convenient glass vessel fitted with electrodes may be used. A suitable set-up has been made using for the sample holder a 2-oz. screw-cap type bottle fitted with a rubber stopper with two brass electrodes ( $\frac{1}{8}$  to  $\frac{1}{4}$ -in. rod) and a glass tube outlet for the gases. Arc-formed gases shall be led (in series with the glass wool filter) to the gas collection and measuring burette. After clamping in place, the vessel shall be filled to three fourths of its capacity with the liquid to be arced.

(b) Electric current (110 v.d.c.) shall then be connected externally to the electrodes. The line should contain a resistance of 4 to 5 ohms. Arcing shall be carried out by manual make-and-break of the electrodes in the liquid. The external portion of the electrodes should be insulated.

(c) Analysis of the sample for carbon dioxide, unsaturated hydrocarbons, oxygen, carbon monoxide, saturated hydrocarbons, and hydrogen shall be carried out in the usual manner.

(d) The gas shall first be collected over trichlorobenzene or carbon tetrachloride which has been saturated with hydrogen chloride gas. The first absorption pipette of the gas analyzer shall then be filled with water and the halogen-containing gas determined by the percentage of gas absorbed at this stage. The residue shall then be submitted to the regular analysis for the gases previously mentioned. In order that no combustible gases may pass undetected, the explosion test for hydrogen and the saturated hydrocarbons should be carried out with oxygen.

### Precautions

28. (a) Before collecting gases for analysis, the connecting tubing of the apparatus shall be flushed out using a

total of not less than 100 ml. of the arc-formed gases.

(b) Check tests shall be made on fresh samples of the liquid.

### REFRACTIVE INDEX

#### Apparatus

29. The apparatus shall consist of the following:

(a) *Refractometer*.—An Abbé refractometer with scale graduated directly in terms of refractive index of the D line of sodium at a temperature of 20 C.

(b) *Water Supply*.—A water supply which may be varied as to temperature.

#### Procedure

30. (a) The refractometer shall be placed in front of a suitable source of light (either daylight or electric light), the thermometer inserted and circulation of water adjusted so as to bring the prisms to the desired temperature (usually 20 C.). A drop of the liquid to be tested shall then be spread upon the upper prism after which the lower prism shall be turned over and clamped. The mirror shall then be adjusted so that the light enters the telescope. The eyepiece shall be focussed on the cross-hairs and the reading lens on the scale. Upon moving the prism arm, a position can now be found where the lower part of the field is dark and the upper part light. In general, the border line will be colored and this is corrected by turning the milled head on the right of the telescope so that a sharp black and white edge is obtained. The prism arm is now moved until this black edge just crosses the intersection of the cross-hairs. The refractive index shall then be read off on the scale, the fourth decimal place being estimated.

(b) The accuracy of the instrument may be checked by a small test plate of known refractive index which is sup-

plied with the refractometer. This test plate shall be attached to the upper prism with a liquid of high refractive index (usually mono-brom naphthalene). Errors may be corrected by means of a small adjusting screw on the back of the telescope.

#### DIELECTRIC STRENGTH

NOTE.—A method for determining dielectric strength of askarels is in preparation by the committee. Meanwhile, reference should be

made to the method of test for liquid dielectrics in the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149).<sup>3</sup>

#### POWER FACTOR

NOTE.—A method for determining power factor of askarels is in preparation by the committee. Meanwhile, reference should be made to the method of test for liquids in the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>



# *Tentative Method of Test for*

## INORGANIC CHLORIDES AND SULFATES IN INSULATING OILS<sup>1</sup>



A.S.T.M. Designation: D 878-46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is intended for the qualitative determination of inorganic chlorides and sulfates in electrical insulating oils.

NOTE.—It should be pointed out that if the strong acids, hydrochloric and sulfuric, are present, they will give positive results for chlorides and sulfates, respectively.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Separatory Funnel*, 250 ml.

(b) *Beaker*, 250 ml.

(c) *Test Tubes*, approximately 25 by 150 mm.

### Reagents

3. The following reagents are required:

(a) *Silver Nitrate Solution* (10 per cent).

(b) *Barium Chloride Solution* (10 per cent).

(c) *Nitric Acid*, c.p. (sp. gr. 1.42).

(d) *Hydrochloric Acid*, c.p. (sp. gr. 1.18).

### Procedure

4. (a) In the 250-ml. separatory funnel, shake thoroughly 100 ml. of the oil with 75 ml. of distilled water at 95 C. Allow to separate, then draw off the water layer into the 250-ml. beaker.

(b) Pour 15 ml. of the separated water layer into a clean test tube, add a few drops of nitric acid (sp. gr. 1.42), and then add 5 ml. of silver nitrate (10 per cent). The appearance of a white precipitate indicates the presence of chlorides.

(c) Pour a second portion of 15 ml. of the water into a clean test tube. Heat to the boiling point, add 5 drops of hydrochloric acid (sp. gr. 1.18), and then slowly add 5 ml. of barium chloride (10 per cent). A white precipitate reveals the presence of sulfates.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

# *Tentative Method of Test for*

## DIELECTRIC STRENGTH OF INSULATING OIL OF PETROLEUM ORIGIN<sup>1</sup>



A.S.T.M. Designation: D 877 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of testing electrical insulating oils applies to oil of petroleum origin for use in transformers, oil circuit breakers, and similar apparatus, as an insulating or cooling medium, or both.

### Apparatus

2. (a) *Transformer*.—The desired test voltage may be most readily obtained by a step-up transformer energized from a variable low-voltage source. The transformer and its controlling equipment shall be of such size and design that, with the test specimen in circuit, the crest factor (ratio of maximum to mean effective) of the test voltage shall not differ by more than plus or minus 5 per cent from that of a sinusoidal wave over the upper half of the range of test voltage. The crest factor may be checked by means of an oscillograph, or a sphere-gap or peak-reading voltmeter in conjunction with a rms. voltmeter. Where the waveform cannot be determined conveniently,

a transformer having a rating of not less than 2 kva. shall be used. Tests shall be made at commercial power frequencies.

NOTE.—When a transformer is used at voltages lower than its full rating, the current drawn from the high voltage winding should not exceed the full-load full-voltage current rating.

(b) *Circuit Breaker*.—The test transformer circuit shall be protected by an automatic circuit-breaking device designed to open substantially instantaneously on the current produced by breakdown of the test specimen. A prolonged flow of current at the time of breakdown causes carbonization of the liquid and pitting and heating of the electrodes, and thereby increases the work of electrode and test cup maintenance, and time of testing.

(c) *Voltage Control*.—The rate of voltage rise shall not, for short-time tests, vary more than plus or minus 25 per cent from the specified rate. Control of voltage may be secured in one of several ways:

- (1) variable-ratio auto-transformer,
- (2) resistance-potential divider,
- (3) generator-field regulation,

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

- (4) induction regulator, or
- (5) adjustable resistance in the primary circuit.

Preference should be given to equipment having an approximately straight-line voltage-time curve over the desired operating range. Motor drive, with variable speed control, should be preferred to manual drive because of the difficulty of maintaining a reasonably uniform rate of voltage rise with the latter.

(d) *Voltmeter*.—The voltage shall be measured by an approved method which gives root-mean-square values, preferably by means of:

- (1) A voltmeter connected to the secondary of a separate potential transformer,
- (2) An electro-static voltmeter in the high-voltage circuit, or
- (3) A voltmeter connected to a well-designed tertiary coil in the test transformer.

A voltmeter connected to the low-voltage side of the testing transformer may be used only if the ratio of transformation does not change appreciably with load.

(e) *Rate of Rise of Voltage*.—The rate of voltage rise may be calculated from measurements of time required to raise the voltage between two prescribed values. When motor-driven regulating equipment is used, the speed-control rheostat may be calibrated in terms of voltage rise for any particular test transformer.

### Electrodes

3. The electrodes shall be polished metal disks 1 in. in diameter with square edges, mounted with axes horizontal and coincident.

### Test Cup

4. A special test cup with the electrodes rigidly mounted with their axes in

a horizontal line shall be used. The test cup should be of such design that no part of it is less than  $\frac{1}{2}$  in. from any part of the test faces of the electrodes, when the latter are in the testing position. The total leakage and charging current of the test cup, when filled with a good grade of insulating oil, shall be less than 200 microamperes at 20 kv., 60 cycles. The test cup shall be made of a material of high dielectric strength, must be insoluble in, and unattacked by, any of the cleaning or test liquids, and non-absorbent to moisture, and the cleaning and test liquids. Wood is not a suitable material for the test cup. The top of the cup should be about  $1\frac{1}{4}$  in. (33 mm.) above the top of the electrodes.

### Adjustment and Care of Electrodes and Test Cup

5. (a) The electrodes and the test cup shall be wiped clean with dry, calendered tissue paper or with a clean, dry chamois skin. The spacing of the electrodes of the test cup shall be checked with a standard round gage having a diameter of 0.100 in. or flat steel "go" and "no-go" gages having thicknesses of 0.0995 and 0.1005 in., respectively, and the electrodes locked in position. It is important to avoid touching the electrodes or the cleaned gage with the fingers or with portions of the tissue paper or chamois skin which have been in contact with the hands. The electrodes and cup shall then be rinsed with dry lead-free gasoline, Stoddard solvent, or dry, water-white kerosine until they are entirely clean. Care shall be taken not to touch the electrodes or the inside of the cup after cleaning, so as to avoid any possible contamination. After thoroughly cleaning the test cup, it shall be filled with a sample of the cleaning fluid and voltage applied with uniform increase at the rate of approximately 3 kv. per sec. (rms. value) until breakdown occurs.

If the breakdown is not less than the established value of the oil being tested, the cup shall be considered in suitable condition for testing. If a lower value is obtained, the cup shall again be thoroughly cleaned and the test repeated. A cleaning fluid whose breakdown is not less than the established value of the oil being tested must be used.

(b) At the beginning of each day's testing, the electrodes shall be examined for pitting and carbon accumulation, and the spacing checked. The cup shall be thoroughly cleaned and tested as described in Paragraph (a). It shall then be flushed with a portion of the sample to be tested before it is filled for the test.

(c) If the test of a sample is below the breakdown value being used by the operator as a minimum satisfactory value, the cup shall be cleaned and prepared as described in Paragraph (a) before testing the next sample.

NOTE.—Evaporation of the cleaning fluid from the electrodes may chill them sufficiently to cause moisture to condense on their surfaces. For this reason, after the final rinsing with cleaning fluid, the cup must immediately be flushed with the oil to be tested, following which the cup should be filled for the test.

## Procedure

6. (a) *Preparation of Sample*.—The dielectric strength of liquid dielectrics may be markedly altered by the migration of impurities through the liquid. In order that representative test specimens may be obtained, the container shall be gently tilted or inverted and the oil swirled several times before each filling of the test cup, in such a way that any impurities present will be thoroughly mixed with the dielectric. Too rapid agitation is undesirable, as it introduces an excessive amount of air into the mixture. Immediately after mixing, the test cup shall be filled with oil to a height of not less than 20 mm. (0.787 in.)

above the top of the electrodes. In order that entrapped air may escape, the container shall be gently rocked a few times and the oil allowed to stand in the cup for 3 min. before voltage is applied.

(b) *Temperature of Test*.—The temperature of the sample when tested shall be the same as that of the room, but the room temperature shall in no case be less than 20 C. (68 F.). Testing at oil temperatures lower than room temperatures is likely to give variable results which may be misleading.

(c) *Application of Voltage*.—Voltage shall be applied and increased at a uniform rate of 3 kv. per sec. from zero until breakdown occurs as indicated by a continuous discharge across the gap. (Occasional momentary discharges which do not result in a permanent arc may occur; these should be disregarded.)

## Number of Tests

7. (a) Except as specified in Paragraph (b), one breakdown test shall be made on each of five fillings of the test cup. If the average deviation from the mean exceeds 10 per cent or if any individual test deviates more than 25 per cent from the average, additional tests shall be made. The dielectric strength shall be determined by averaging the first five tests that conform to the allowable variations.

(b) When oil is tested in considerable quantity, so that the time required for testing in accordance with Paragraph (a) is excessive, and when it is merely desired to determine whether the breakdown safely exceeds the limit specified, or in those cases where the amount of oil available for test may be very limited, one breakdown test shall be made on each of two fillings of the test cup. If neither breakdown is below this value, the oil may be considered satisfactory and no further tests shall be required. If either of the two breakdowns is less than

the specified value a breakdown shall be made on each of three additional fillings and test results analyzed in accordance with Paragraph (a).

#### Report

8. The report shall include the following:

(1) Procedure used, whether Paragraph (a) or (b), Section 7.

(2) The volts (rms. value) at each breakdown and the average of the five breakdowns. All results shall be reported to the nearest kilovolt.

(3) The approximate temperature of the oil at the time of the test.

# *Tentative Methods of Test for*

## GAS CONTENT OF INSULATING OILS<sup>1</sup>



A.S.T.M. Designation: D 831 - 45 T

ISSUED, 1945.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures. for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods of test are intended for determining the gas content of electrical insulating oils of low viscosities of the general order of 100 sec. Saybolt Universal at 100 F., such as are used in capacitors and paper insulated lead-covered electric cables of the oil-filled type. The determination of gas content is desirable for any insulating oil of these properties intended for use in a degassed state. Two methods are covered: a routine method, and a referee method for use when a test of high accuracy is desired (see Explanatory Note).

### ROUTINE METHOD

#### Nature of Test

2. The routine method consists essentially of feeding the oil into an evacuated chamber in such a manner that the oil is thoroughly exposed to the vacuum, allowing free escape of any dissolved gas. From the volume of oil admitted to the chamber, the temperature, the pressure produced, and volume occupied by the released gas, the gas volume under standard conditions of pressure and

temperature may be calculated as a percentage by volume of oil.

#### Sampling

3. (a) When convenient, the degassing chamber of the measuring equipment shall be connected directly to the container from which the oil is to be sampled. This is usually not convenient and is often impossible. The method of sampling described in Paragraph (b) is recommended as an alternate (Note).

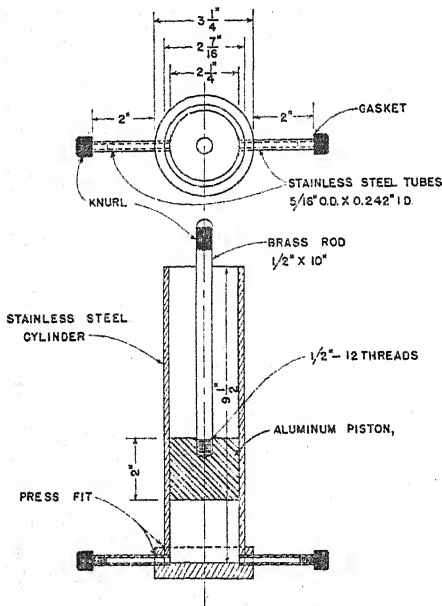
NOTE.—This sampling method has been in use by two laboratories for many years.

(b) The sample container shown in Fig. 1 shall consist of a stainless steel cylinder  $2\frac{1}{4}$  in. in inside diameter and  $9\frac{1}{2}$  in. in length, closed at the bottom. An aluminum piston shall be inserted in the bore of the cylinder accurately machined for an easy sliding fit. Two nipples, diametrically opposed, shall be inserted at the extreme bottom of the cylinder. Each nipple shall have a screw plug at the end with a gasket for sealing. All connections to the measuring equipment from the sample container should be of glass or metal tubing. Butted joints connected by short sections of heavy-walled rubber tubing may be used provided the tubing is thoroughly coated

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

with suitable sealing compound. All samples shall be taken under slight oil pressure, with the following sequence of operations: Push the piston to the extreme bottom of the cylinder. Remove plugs from both nipples. Hold the cylinder so that the nipples point in a vertical direction. By means of a rubber tube connection, force oil in through the nipple in the lowest position and flush a few milliliters out the opposite nipple to remove any trapped air bub-



NOTE.—The aluminum piston shall have a diametrical clearance of 0.0005 to 0.0010 in.

FIG. 1.—Apparatus for Sampling Oils for Determination of Gas Content.

bles. Then insert the plug in the outlet nipple and allow oil to push the piston up to fill the cylinder. Hold the piston at the top of the cylinder with one hand and plug the inlet nipple. The aluminum piston "floats" on the oil as the level varies, due to temperature changes or removal of test samples and prevents contamination by absorption and diffusion. The piston shall be accurately fitted to move down freely with decreasing oil level to prevent voids forming under the piston which would

allow rapid absorption of air by the top oil. When drawing the test sample continuously from the cylinder, it is advisable to weight the piston to insure maintenance of contact with the oil. Wide variations in the results are possible in two samples from the same source unless the greatest care is taken in the sampling

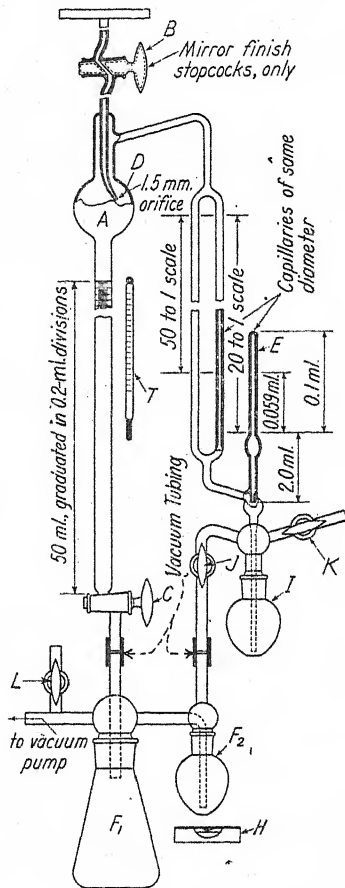


FIG. 2.—Apparatus for Routine Determination of Gas Content of Insulating Oil.

procedure. This phase of the test is so involved with the details of what constitutes correct practice that ability to procure consistent representative samples depends, to a great extent, on wide experience. The chief precaution to insure procurement of representative samples involves a complete flushing of all piping and hose between the sample

container and sample source, such as pothead, joint, cable, oil reservoir, etc., immediately preceding collection of the sample. All long pipe lines should be eliminated where possible. After taking the sample, it is very important to see that the piston always remains in contact with the oil prior to and during withdrawal of the test specimen.

### Apparatus

4. The apparatus, shown in Fig. 2, shall consist of the following:

(a) *Degassing Chamber*.—Degassing chamber, *A*, made of heat-resistant glass<sup>3</sup> (with calibrated oil well at bottom), having fixed total space volume of about 175 ml. The oil well shall have a maximum capacity of 50 ml. and shall be calibrated in 0.2-ml. divisions.

(b) *Stopcocks*.—Glass stopcocks *B*, *C*, *J*, *K*, and *L*, which shall have large diameter barrels and a mirror finish to insure against leakage. Stopcock grease<sup>4</sup> shall be used on all stopcocks and ground-glass joints.

(c) *Pipette*.—Glass pipette or "atomizer", *D*, placed to drop oil on side of degassing chamber.

(d) *Pressure Gage*.—Pressure gage, *E*, of modified McLeod type marked for two scale factors, 50 to 1 and 20 to 1. This will provide ranges sufficient for most testing. If a higher pressure range is desired, a separate complete equipment using an absolute mercury manometer in place of the McLeod gage may be used.

(e) *Oil Traps*.—Two oil traps, *F*<sub>1</sub> and *F*<sub>2</sub>.

(f) *Thermometer*, *T*.

(g) *Level*.—Spirit level, *H*, normal to the vertical axes of mercury columns.

(h) *Mercury Well*, *I*.

### Calibration of Apparatus

5. Calibration and conditioning of the apparatus is often done by the manu-

facturer. If this has not been done, or if a check is desired, the following procedure may be used:

(a) Clean the glass assembly with a cleaning solution, wash with distilled water, and dry. Weigh the empty tester with slight film of grease on the stopcocks. Record weight in grams as *W*. Fill with distilled water and weigh. Record weight in grams as *R*. The difference between *W* and *R* is the volume of the tester in milliliters. Correction for temperature of water may be made, but is an unnecessary refinement.

(b) Empty and thoroughly dry equipment in oven. Pour the mercury for the McLeod gage into the mercury well, grease the stopcocks and ground-glass joints and assemble the apparatus. Attach to the mounting board (with spirit level horizontal) and align with a plumb bob and line.

### Procedure

6. (a) Sample and apparatus should be at room temperature (20 to 30 C.).

(b) Evacuate the degassing chamber with stopcock *B* closed and stopcock *C* open.

NOTE.—These stopcocks must be accurately ground and kept well greased to prevent leaks.

(c) When the McLeod gage reading is reasonably constant and less than 0.1 mm. (absolute pressure) close stopcock *C* for about 15 min. to test for any leaks. No observable pressure change should occur in this time. (Let vacuum pump run continuously to enhance seal of stopcock *C*.)

(d) Flush oil slowly from sample through stopcocks *B* and *C* so as to thoroughly wash walls of glass tubing and to remove any trapped air bubbles in system above stopcock *B*. (About 50 ml. of oil are commonly used for this flushing.)

(e) Reevacuate the degassing chamber, close stopcock *C* and by careful manipulation of stopcock *B* feed oil

<sup>3</sup> Pyrex glass is satisfactory for this purpose.

<sup>4</sup> A stopcock grease equivalent to No. 15521A Vacuum Sealing Compound, Central Scientific Co., Chicago, Ill., has been found satisfactory for this purpose.



from the sample container into the degassing chamber at a very slow rate so that the oil falls in single drops from pipette *D*. This rate will generally average 1 drop per second or less. The oil should show no appreciable tendency to bubble or foam after falling into the oil well at the bottom of the degassing chamber. If this occurs the rate of flow should be decreased.

(f) When 25 ml. of oil have been degassed, close stopcock *B* and read the McLeod gage and thermometer. In reading the gage the mercury level shall be adjusted by manipulation of stopcocks *J* and *K*.

(g) Empty the oil reservoir into the oil trap, and complete the evacuation of the degassing chamber for the next test.

#### Calculation

7. The gas content shall be calculated as follows:

$$G = \frac{35.9[(V - V_1)P_2 - VP_1]}{(273 + t)V_1}$$

where:

*G* = gas content (at 760 mm. of mercury and 0 C.) expressed as a percentage by volume of oil,

*V* = known total fixed volume of apparatus in milliliters,

*V*<sub>1</sub> = volume of oil in milliliters,

*P*<sub>1</sub> = initial pressure of gas in chamber, in millimeters of mercury,

*P*<sub>2</sub> = final pressure of gas in chamber, in millimeters of mercury,

*t* = temperature of chamber, in degrees Centigrade. (The room temperature shall be read from thermometer, *T*.)

#### REFEREE METHOD

##### Nature of Test

8. The referee method consists essentially of feeding the oil into an evacuated

chamber in such a manner that the oil is thoroughly exposed to the vacuum, allowing free escape of any dissolved gas. From the volume of oil admitted to the chamber, the temperature, the pressure produced, and the volume occupied by the released gas, the gas volume under standard conditions of pressure and temperature may be calculated as a percentage by volume of oil.

#### Sampling

9. The oil shall be sampled as described in Section 3.

#### Apparatus

10. The apparatus, shown in Fig. 3, shall consist of the following:

(a) *Degassing Chamber*.—Degassing chamber, *A*, made of heat-resistant glass<sup>3</sup> (with calibrated oil well at bottom), having fixed total space volume of about 175 ml. The oil well shall have a maximum capacity of 50 ml. and shall be calibrated in 0.2-ml. divisions.

(b) *Stopcocks*.—Glass stopcocks, *B*, *C*, *L*, *M*, *N*, which shall have large diameter barrels and a mirror finish to insure against leakage. Stopcock grease<sup>4</sup> shall be used on all stopcocks and ground-glass joints.

(c) *Pipette*.—Glass pipette or "atomizer," *D*, placed to drop oil on side of degassing chamber.

(d) *Pressure Gage*.—Pressure gage, *E*, of modified McLeod type marked for two scale factors, 50 to 1 and 20 to 1. This will provide ranges sufficient for most testing. If a higher pressure range is desired, a separate complete equipment using an absolute mercury manometer in place of the McLeod gage may be used for the high pressures.

(e) *Oil Traps*.—Two oil traps, *F*<sub>1</sub> and *F*<sub>2</sub>.

(f) *Thermometer*, *T*.

(g) *Level*.—Spirit level, *H*, normal to the vertical axes of mercury columns.

(h) *Mercury Well*, *I*.

(i) *Cold Trap*, *J*.

(j) *Oven*.—An oven, *K*, enclosing the degassing chamber, *A*, provided with suitable means for reading, maintaining, and regulating temperature in a range from 30 to 150 C. Temperature shall be measured by means of a thermocouple fastened to the oil chamber at the

(a) Clean the glass assembly with a cleaning solution, wash with distilled water, and dry. Weigh the empty tester, with a slight film of grease on the stopcocks, and record the weight in grams as *W*. Fill with distilled water, weigh, and record the weight in grams as *R*. The

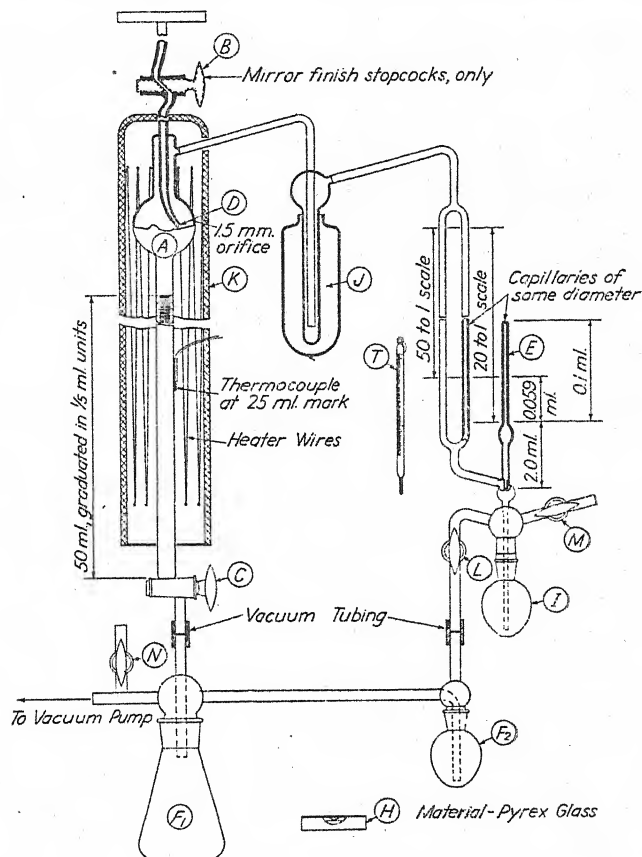


FIG. 3.—Apparatus for Referee Determination of Gas Content of Insulating Oil.

25-ml. mark and suitably shielded to eliminate radiation errors.

#### Calibration of Apparatus

11. Calibration and conditioning of the apparatus is often done by the manufacturer. If this has not been done, or if a check is desired, the following procedure may be used:

difference between *W* and *R* is the volume of the tester in milliliters. Correction for temperature of water may be made, but is an unnecessary refinement.

(b) Empty and thoroughly dry the equipment in an oven. Pour the mercury for the McLeod gage into the mercury well, grease the stopcocks and ground-glass joints and assemble the

apparatus. Attach to the mounting board (with spirit level horizontal) and align with a plumb bob and line.

(c) Calculate as closely as possible the separate volumes of the degassing chamber,  $V_d$ , enclosed in the oven, and the section of the cold trap,  $V_t$ , enclosed in the vacuum flask.

### Procedure

12. (a) Regulate the temperature of the oven to 50 C. and make certain that it remains constant within plus or minus 2 C. for at least 30 min.

(b) Fill the vacuum flask surrounding the cold trap,  $J$ , with either liquid air or carbon dioxide snow, or equivalent.

(c) The sample and apparatus shall be at room temperature (20 to 30 C.) with the exception of the parts mentioned under Paragraphs (a) and (b).

(d) Evacuate the degassing chamber with stopcock  $B$  closed and stopcock  $C$  open.

NOTE.—These stopcocks must be accurately ground and kept well greased to prevent leaks.

(e) When the McLeod gage reading is reasonably constant and less than 0.1 mm. (absolute pressure), close stopcock  $C$  for about 15 min. to test for any leaks. No observable pressure change should occur in this time. (Let vacuum pump run continuously to enhance seal of stopcock  $C$ ).

(f) Flush oil slowly from the sample through stopcocks  $B$  and  $C$  so as to wash thoroughly the walls of the glass tubing and to remove any trapped air bubbles in the system above stopcock  $B$ . (About 50 ml. of oil are commonly used for this flushing).

(g) Reevacuate the degassing chamber, close stopcock  $C$ , and by careful manipulation of stopcock  $B$  feed oil from the sample container into the degassing chamber at a very slow rate so

that the oil falls in single drops from pipette  $D$ . This rate will generally average one drop per second or less. The oil should show no appreciable tendency to bubble or foam after falling into the oil well at the bottom of the degassing chamber. If this occurs, the rate of flow should be decreased.

(h) When 25 ml. of oil have been degassed, close stopcock  $B$  and read the McLeod gage, the temperature of the room and of the degassing chamber. The temperature of the cold trap  $t_i$  will be known and will depend, of course, upon the coolant used. (Temperature of liquid air is  $-194$  C., and of carbon dioxide snow  $-78$  C.). In reading the McLeod gage the mercury level shall be adjusted by manipulation of stopcocks  $L$  and  $M$ .

(i) Empty the oil reservoir into the oil trap, and complete the evacuation of the degassing chamber for the next test.

### Calculation

13. The gas content shall be calculated as follows:

$$G = \frac{35.9}{V_1} \left[ \frac{(V - V_d - V_t)(P_2 - P_1)}{273 + t_a} + \frac{(V_d - V_t)P_2 - V_d P_1}{273 + t_d} + \frac{V_t(P_2 - P_1)}{273 + t_i} \right]$$

where:

$G$  = gas content (at 760 mm. of mercury and 0 C.) expressed as a percentage by volume of oil,

$V$  = known total fixed volume of apparatus in milliliters,

$V_1$  = volume of oil in milliliters,

$V_d$  = volume of degassing chamber in milliliters,

$V_t$  = volume of cold trap in milliliters,

$P_1$  = initial pressure of gas in chamber in millimeters of mercury,

$P_2$  = final pressure of gas in chamber in millimeters of mercury,

$t_a$  = ambient temperature in degrees Centigrade read on thermometer,  $T$ ,

$t_d$  = temperature of degassing chamber  
A in degrees Centigrade, and  
 $t_c$  = temperature of cold trap in degrees Centigrade.

NOTE.—The calculation may appear to be tedious, but when the fixed volumes and temperatures are substituted in the formula, it will be found that it can be simplified considerably. Simplification will also be aided if  $P_1$  can be made low enough to be negligible.

### EXPLANATORY NOTE

NOTE.—There are several possible sources of error in the routine method but due to the convenience in making the test by this procedure, and the simplicity and portability of the test equipment this method is considered satisfactory for routine tests. The possible errors due to accuracy of readings are relatively small for normal ranges of gas content.

Consider the case of an oil with 0.10 per cent gas. For normal procedure, values of initial pressure and end point would be 0.100 and 0.255 mm. of mercury, respectively. This pressure increase would be developed by 25 ml. of oil tested at 25 C. (in a degassing chamber of total fixed space volume of approximately 175 ml.).

Pressures can be read to plus or minus 0.004 mm. of mercury. (This is equivalent to reading the mercury columns of the gage to plus or minus 0.20 mm. assuming a 50 to 1 scale factor of the gage). Oil volume can be read to plus or minus 0.05 ml.

Assuming errors in pressure and volume readings adding to give maximum deviation, the resultant error would be plus or minus 0.0067 in 0.10 per cent, equivalent to a percentage error of 6.7 per cent for gas contents of this low order.

The above analysis also applies to the referee method. The only difference would be due to the different temperatures used and the correction would be negligible.

Attention, however, is called to the following

possible errors present in this apparatus: It is well known that the McLeod gage gives correct pressure readings only for pure gases. If vapor is present, the gage will give entirely erroneous readings, due to the high-pressure ratios present between the two columns of mercury, and consequent condensation of vapor in the high-pressure side. This would cause the pressure reading to be low with a resultant low gas content determination.

In the referee method this possibility of error is eliminated by the use of a cold trap to freeze out any such vapors which may be removed from the oil. This also allows the use of a higher temperature in the degassing chamber, thereby lowering the viscosity of the oil under test and increasing the ease of removal of gas.

Attention should also be called to the possibility of gas leakage through any rubber tubing which may be present in the sample supply line. This may occur in spite of positive oil pressure being maintained in this line, causing, of course, higher gas content of the sample.

Also, unless sampling is done with extreme care by an experienced operator, an increase in the gas content of the sample may very easily occur, causing a high reading of gas content. Tests have also shown that all of the gas may not be entirely removed from the oil at room temperature. This error may amount to as much as 50 per cent, and, of course, results in low readings of gas content.

# *Tentative Methods of Test for*

## SLUDGE FORMATION IN MINERAL TRANSFORMER OIL<sup>1</sup>



A.S.T.M. Designation: D 670 - 42 T

ISSUED, 1942.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods of test are applicable in the evaluation of the sludge forming tendency in new, mineral transformer oils (see Explanatory Note). Two methods are covered as follows:

(a) *Method A*, the sludge accumulation test, is applicable to new, mineral transformer oils except those that have been "stabilized" by the addition of retarders, stabilizers, and negative catalysts.

(b) *Method B*, the high-pressure oxidation test, is applicable to new, mineral transformer oils except the so-called highly refined, white oils of zero iodine value, and mineral oil containing retarders, stabilizers, and negative catalysts.

### METHOD A: SLUDGE ACCUMULATION TEST

#### Nature of Test

2. The sludge accumulation test for mineral transformer oil evaluates the

propensity of an oil to deposit an insoluble sludge under definitely prescribed conditions of oxidation in the presence of a copper catalyst for 72, 168, and 336 hr. The total evaluation of an oil includes consideration of the tendency of the oil to deposit sludge under mild oxidation (72 hr.) and the rate at which this sludge deposition increases as a result of more severe oxidation as the test is continued for 168 and 336 hr.

#### Apparatus

3. The apparatus shall consist of the following:

(a) *Aging (Oxidation) Bath*.—An oil or wax bath of a suitable type capable of controlling the temperature at  $120 \pm 0.5$  C. with a temperature gradient of less than 1 C. in the body of the oil or wax used. Circulation of the oil or wax heating medium such as by a pump or stirrer is recommended.

(b) *Drying Tower*.—A drying tube, 10 to 12 in. (25 to 31 cm.) in height, fitted with a ground-glass stopper and side tubes for conditioning of the air supply.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1942.

(c) *Oil Receptacle and Cover*.—An oil receptacle consisting of a heat-resistant, glass test tube<sup>3</sup> 25 mm. in outside diameter, 1.25 mm. in wall thickness, and 150 mm. in length, with a capacity of 50 ml., equipped with a loosely-fitting glass cover having a central hole to admit the air supply tube.

(d) *Gooch Crucible*. — A porcelain Gooch crucible, size No. 4, 35-ml. capacity, equipped with a standard quantitative filter paper disk.<sup>4</sup>

(e) *Copper Catalyst*. — Commercial copper wire 0.040 in. (1.016 mm.) in diameter.

#### A.S.T.M. Precipitation Naphtha

4. A.S.T.M. precipitation naphtha<sup>5</sup> for dilution of the oxidized oil and precipitation and washing of the sludge shall conform to the following requirements:

Test	Limit	A.S.T.M. Designation <sup>a</sup>
Gravity, deg. A.P.I. at 60 F....	70 to 73	D 287
Aniline point.....	58 to 60 C.	D 611
Initial boiling point.....	not less than 122 F. (50 C.)	D 86
50 per cent point.....	158 to 176 F. (70 to 80 C.)	D 86
End point.....	not more than 266 F. (130 C.)	D 86

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287).<sup>4</sup>

Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611).<sup>6</sup> and

Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86).<sup>6</sup>

#### Preparation of Air Supply

5. The oxidation of the oil shall be obtained by contact with room air conditioned as follows:

<sup>3</sup> Pyrex glass is satisfactory for this purpose.

<sup>4</sup> A suitable quantitative filter paper disk may be obtained from Carl Schleicher & Schuell, designated as white ribbon.

<sup>5</sup> These requirements for precipitation naphtha are identical with those prescribed in Section 4 of the Standard Method of Test for Precipitation Number of Lubricating Oils (A.S.T.M. Designation: D 91), see p. 257.

<sup>6</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

(a) Dry the air by forcing it through a solid desiccant of high moisture absorbing capacity.<sup>7</sup> Arrange the desiccant in the drying tower to a depth of 8 to 10 in. Change the desiccant frequently (Note).

NOTE.—Desiccants should not be used for a period longer than 1 week.

(b) After passing the air through the drying tower, admit it directly to the receptacle containing the oil to be tested. Do not preheat the air.

(c) Determine the rate of air supply with a flow meter, and adjust the flow to deliver the conditioned air at a rate of  $0.5 \pm 0.1$  liter per hour to each tube of oil being tested.

(d) Rubber connections shall be eliminated as much as possible and when used, the rubber shall be at room temperature. No rubber connections shall be used in contact with the oil or wax of the heating bath or under conditions where the temperature of the rubber will be raised above room temperature. Where it is necessary to use rubber connections, only sulfur-free rubber is permissible.

#### Preparation of Oil Receptacle

6. Wash each oil receptacle thoroughly first with acetone, then with soap and water, and rinse in chromic acid solution. Wash each receptacle free from chromic acid using tap water, and finally rinse with distilled water. Dry in an air oven at 105 to 110 C. for at least 3 hr., cool to room temperature in a desiccator, and keep the receptacle in the desiccator until ready for use.

#### Preparation of Copper Catalyst

7. (a) Immediately before use, polish the copper wire with No. 00 emery cloth or its equivalent and wipe free from abrasive with a clean dry cloth.

(b) Wind a 12-in. (30.5-cm.) length of the polished wire into a helical coil

<sup>7</sup> Dehydrite is a suitable desiccant for this purpose.

approximately 22 mm. in outside diameter and 2 in. (5.08 cm.) in length. Clean the coil thoroughly with c.p. ethyl ether, air-dry, and insert immediately into the oil receptacle. Handle the clean copper coil only with tongs in order to avoid contamination.

### Preparations of Gooch Crucible

8. (a) Cover the bottom of the Gooch crucible with the filter paper disk.

(b) Prepare a slurry for the formation of the asbestos mat in the Gooch crucible by making a suspension of medium asbestos fiber, Gooch grade, in the proportion of 20 g. of asbestos to 1 liter of distilled water. Pour the asbestos suspension into the Gooch crucible, tamp with a glass rod, and dry by suction. In this manner build up an asbestos mat to a depth of approximately  $\frac{1}{16}$  in. (1.59 mm.). Then pour into the crucible, without tamping, additional asbestos suspension to build up the mat to a total thickness of approximately  $\frac{3}{8}$  in. (2.17 mm.) after drying by suction.

(c) Place the prepared crucible in an air oven and dry at 105 to 110 C. for 15 to 24 hr., cool in a desiccator, and when needed, weigh to the nearest 0.0001 g.

### Conditioning of Sample

9. Filter by gravity the oil to be tested using a previously dried, acid-free filter paper to remove traces of sediment, fibers, and excess water. (Dry the filter paper for 1 hr. at 100 C. before using.) Discard the first 25 ml. of the oil filtrate. Carefully protect the filtrate during the filtration against dust and other contamination.

### Procedure

10. (a) Prepare three copper catalysts as described in Section 7, insert one each in a clean, dry oil receptacle prepared

as described in Section 6, and weigh to the nearest 0.0001 g.

(b) Transfer 25 ml. of the conditioned oil sample into each of the three prepared oil receptacles by means of a clean, dry pipette. Protect the oil from contamination by atmospheric moisture and dust during the interval between the placing of the oil in the receptacle and the actual start of the oxidation. Weigh each assembly containing the oil to the nearest 0.0001 g.

(c) Adjust the heating bath to maintain a temperature of  $120 \pm 0.5$  C. during the oxidation of the sample.

### Oxidation

11. (a) Immerse the oil receptacle in the heating bath which has been adjusted to a temperature of  $120 \pm 0.5$  C. Immerse the oil receptacle in the bath to the extent that the surface of the heating bath will be slightly higher than the surface of the test oil in the receptacle. Insert through the holes in the receptacle cover, the clean, dry glass tubes through which the conditioned air is to be led into the oil receptacle. Adjust the tube covers and place the air supply tubes so that they are approximately  $\frac{1}{2}$  in. (1.3 cm.) above the oil level in the receptacle and start the air flow through the receptacle. The air shall not be bubbled through the oil. When more than one oil receptacle is fed from a single air supply line, adjust the air flow to each oil receptacle to a rate of  $0.5 \pm 0.1$  liter per hour.

(b) Oxidize the three oil samples for periods of 72, 168, and 336 hr., respectively, under the carefully controlled conditions prescribed.

### Treatment of Oxidized Oil

12. (a) After the required period of oxidation, shut off the air supply and remove the oil receptacle and sample from the oxidizing bath. Cool for



1 hr. to room temperature in a clean dark box or dark room, protecting the oil sample from dust, moisture, or other contamination, and light.

(b) At the end of the cooling period, remove the copper catalyst from the oil receptacle and scrape off any sludge adhering to the coil, returning the sludge to the oxidized oil in the receptacle. Wash the copper-wire coil with the petroleum naphtha and transfer the washings to the oxidized oil in the receptacle.

(c) Transfer the oxidized oil and naphtha washings to a clean beaker and dilute to 200 ml. at room temperature with the petroleum naphtha. Carefully stir the oil-naphtha mixture until the oil is completely dissolved, cover the beaker, and allow to stand for 1 hr. at a temperature of  $25 \pm 2$  C.

#### Weighing of Sludge

13. (a) Transfer the oil-naphtha solution to the prepared, dried, and weighed Gooch crucible. Also wash any sludge adhering to the oil receptacle free from oil with naphtha, dry and scrape free, and transfer to the crucible by means of a suitable brush. Determine the weight of any sludge that cannot be removed from the oil receptacle from the difference in weight of the sludged receptacle (cleaned to remove bath oil or wax, and dirt from the outside) and the weight of the receptacle after cleaning free from sludge as described in Section 6. Add the weight of the sludge remaining in the receptacle to the weight of the sludge in the Gooch crucible.

(b) Wash the sludge in the Gooch crucible thoroughly with naphtha to remove all traces of adhering oil. Normally three washes of 25-ml. portions each are sufficient. A drop of the final naphtha washing should show no oil stain on the filter paper.

(c) Dry the sludge in the Gooch cru-

cible by suction and place in an air oven at 105 to 110 C. for 1 hr., cool to room temperature in a desiccator, and weigh to the nearest 0.0001 g.

#### Calculation and Report

14. (a) Calculate the percentage of total sludge in the oil as follows:

$$\text{Sludge value} = \frac{\text{wt. of total sludge}}{\text{wt. of oil}} \times 100$$

(b) Report the sludge value for each of the test periods of 72, 168, and 336 hr.

#### METHOD B: HIGH-PRESSURE OXIDATION TEST

##### Nature of Test

15. The high-pressure oxidation test determines the "Bomb Sludge Value," which is the percentage by weight of sludge formed after 24-hr. oxidation at 140 C. under 250 psi. of oxygen pressure.

##### Apparatus

16. The apparatus shall consist of the following:

(a) *Aging (Oxidation) Bath*.—An oil or wax bath of a suitable type capable of controlling the temperature at  $140 \pm 0.5$  C. with a temperature gradient of less than 1 C. in the body of the oil or wax used. Circulation of the oil or wax heating medium such as by a pump or stirrer is recommended. The heat source shall have sufficient capacity to return the bath to the required temperature within 30 min. after immersion of the cold bomb.

(b) *Bomb*.—A pressure bomb of the type shown in Fig. 1, conforming to the dimensions prescribed in Fig. 2. The bomb shall be made of stainless steel, capable of withstanding with safety an internal pressure of 10,000 psi., and provided with a lead gasket for sealing.

(c) *Oil Receptacle*.—An oil receptacle consisting of a heat-resisting, centrifuge-



type tube,<sup>3</sup>  $1\frac{1}{8}$  in. (28.6 mm.) in inside diameter, and 6 in. (152.4 mm.) in length, with a capacity of 100 ml.

(d) *Rack*.—A suitable metallic rack for holding the oil receptacle and its contents in an upright position during the oxidation period.

(e) *Shield*.—An aluminum shield or its equivalent for wrapping around the oil receptacle during the oxidation period to insure cleanliness.

#### A.S.T.M. Precipitation Naphtha

18. The A.S.T.M. precipitation naphtha prescribed in Section 4 shall be used for the dilution of the oxidized oil and the precipitation and washing of the sludge.

#### Preparation of Bomb

19. Clean and dry the high-pressure bomb before using. To insure cleanliness, rinse the bomb with the petroleum

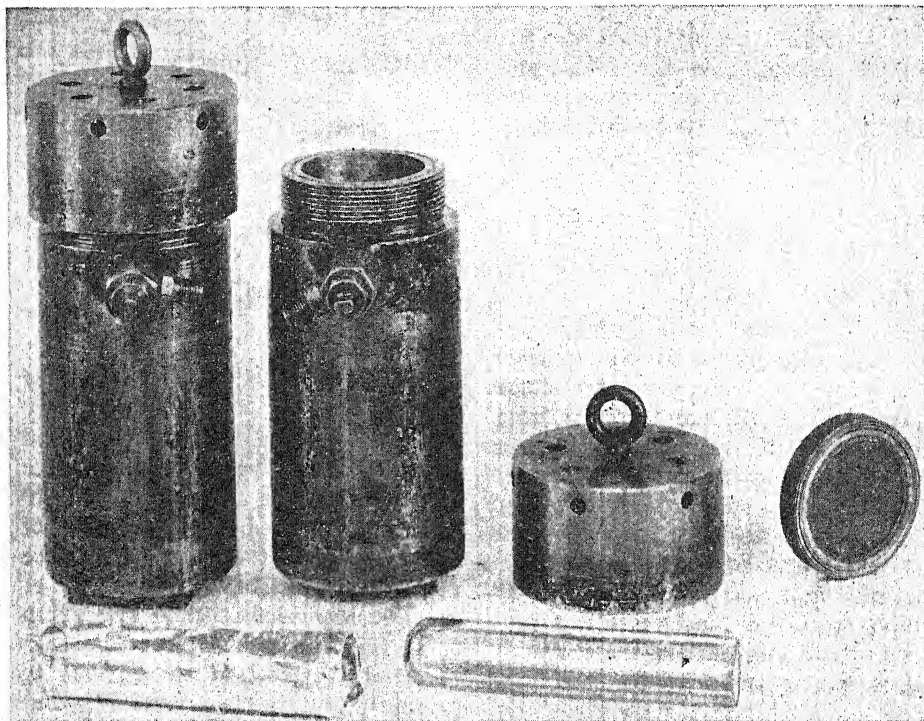


FIG. 1.—Bomb and Apparatus for Determining Bomb Sludge Value of Mineral Transformer Oils.

(f) *Gooch Crucible*. — A porcelain Gooch crucible, size No. 4, 35-ml. capacity, equipped with a standard quantitative filter paper disk.<sup>4</sup>

#### Oxidizing Gas

17. For oxidizing the oil, pure oxygen gas under a gage pressure of 250 psi. at the oxidation temperature of 140 C. shall be used.

naphtha, drain in air, and dry in an air oven at 105 to 110 C. for 3 hr. Cool the bomb to a temperature of  $25 \pm 2$  C. just before using.

#### Preparation of Oil Receptacle

20. Prepare the oil receptacle in accordance with the procedure described in Section 6.



oil in the receptacle and its closure in the bomb. Weigh the prepared receptacle containing oil to the nearest 0.0001 g.

(c) Place the oil receptacle containing the weighed amount of conditioned oil in the bomb in a vertical position using a suitable wire rack or holder. The aluminum shield may be wrapped around the oil receptacle during the oxidation period to insure cleanliness. Set the lead gasket and inside cover in position; then screw on the outside cover. When the bomb has been properly sealed, flush once by filling with oxygen to a pressure of 25 psi. and slowly releasing the pressure to atmospheric pressure.

(d) After flushing, admit the oxygen gas at room temperature to the assembled bomb containing the conditioned oil. Apply the gas pressure so as to produce a gage pressure of 180 psi. at 25 C. This will give the required gas pressure of 250 psi. at the oxidation temperature of 140 C. The oxygen gas must not be applied to the assembled bomb at any temperature above room temperature.

(e) Adjust the heating bath to maintain a temperature of  $140 \pm 0.5$  C. during the oxidation of the sample.

#### Oxidation

25. (a) When the pressure of 180 psi. of oxygen gas in the bomb has been obtained, suspend the bomb immediately in the wax or oil bath which has been adjusted to a temperature of  $140 \pm 0.5$  C. The temperature of the bath will drop when the bomb is immersed. Regulate the heat supply so as to return the temperature to 140 C. within 30 min. after immersion. Totally immerse the charged bomb in the heating bath.

(b) Record the exact time at which the bomb is immersed. Continue the oxidation period for exactly 24 hr.,

maintaining the temperature of the heating bath at  $140 \pm 0.5$  C. (Note).

NOTE.—After the charged bomb has been immersed in the heating bath or its temperature otherwise raised above room temperature, the gas pressure shall not be adjusted or changed in any manner except for unloading the bomb at the end of the oxidation period. No change in gas pressure shall be made while the bomb is immersed in the heating bath. If the gas pressure falls because of leaks, the bomb shall be removed from the heating bath, the gas pressure reduced to atmospheric pressure, the bomb unloaded, and the oil sample discarded. When once the gas pressure has been released, partially or totally, there shall be no reapplication of the gas pressure.

#### Treatment of Oxidized Oil

26. (a) Exactly 24 hr. after the bomb has been placed in the heating bath, remove it from the bath and slowly release the pressure to atmospheric pressure. In order to eliminate erratic results due to gas leakage during the test, check to determine the tightness of the bomb by measuring the volume of gas escaping from the bomb when unloading. The volume of the gas so measured should be within 10 per cent of that expected from the physical volume of the bomb at the average temperature of the gas measured. Tests in which a greater loss of gas has occurred shall be checked. Remove the bomb cover and cool the oil receptacle for 1 hr. to room temperature in a clean dark box or dark room, protecting the oil sample from dust, moisture, or other contamination, and light.

(b) Transfer the oxidized oil to a clean beaker and dilute to 200 ml. at room temperature using the petroleum naphtha. Carefully stir the oil-naphtha mixture until the oil is completely dissolved, cover the beaker, and allow to stand at a temperature of  $25 \pm 2$  C. for 1 hr.

### Weighing of Sludge

27. Weigh the sludge obtained as described in Section 13.

### Calculation and Report

28. (a) Calculate the bomb sludge value as follows:

$$\text{Bomb sludge value} = \frac{\text{wt. of total sludge}}{\text{wt. of oil}} \times 100$$

(b) Report the bomb sludge value as determined.

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### EXPLANATORY NOTE

Investigations under the direction of Subcommittee IV on Liquid Insulation, of Committee D-9 on Electrical Insulating Materials, have shown that the results obtained with either of the tests described in these methods are reproducible if proper care is exercised by the test operator. It must be clearly recognized that to obtain reproducible results the test operator must closely follow the procedure described making certain that the conditions of the oxidation are carefully maintained within the prescribed limits. Variation from the procedure given will frequently give widely divergent test results.

No data are available which correlate the results obtained with the commercial use of transformer oil. In publishing these two test procedures, no implication, direct or implied, is made that either procedure is of commercial operating value. From the comments and criticism obtained as a result of the publication of these test methods, together with the results of the continued study of the methods by the members of Subcommittee IV, Committee D-9,

it is hoped that a true evaluation of their commercial importance may be obtained.

For fundamental details describing each of the test procedures outlined, reference should be made to the following papers: F. M. Clark, and E. A. Snyder, "Testing for Sludge Formation in Mineral Transformer Oil,"<sup>8</sup> and F. M. Clark, "Studies in the Oxidation of Mineral Transformer Oil."<sup>8</sup>

In a general sense, Method A, the Sludge Accumulation Test, is considered of value in studying the sludge forming propensity of a new grade of mineral transformer oil before commercial application. Method B, the High-Pressure Oxidation Test, is considered of value in checking the sludge forming characteristics of a standard grade of oil in order to control the continuity of this property from shipment to shipment. Which type of test is best adapted for determining the condition of mineral oil in transformer use is a decision which must await the results of further study.

<sup>8</sup> *Proceedings, Am. Soc. Testing Mats.*, Vol. 36, Part II, p. 568 (1936), and Vol. 38, Part II, p. 507 (1938), respectively.

# *Tentative Specifications for*

## LOW AND MEDIUM VOLTAGE PIN-TYPE LIME-GLASS INSULATORS<sup>1</sup>



A.S.T.M. Designation: D 730 - 46 T

ISSUED, 1943; REVISED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

*Adapted from Edison Electric Institute Specifications for Low and Medium Voltage Pin-Type Lime-Glass Insulators (TD-51—1942)<sup>3</sup>*

### Scope

1. These specifications cover pin-type lime-glass insulators for mounting on wooden pins or on steel pins equipped with a lead or wooden cob, and metal-bushed lime-glass insulators for mounting on steel or hard-metal threads; both insulator types having a dry flashover of 85,000 v. or less.

### General

2. (a) Unless otherwise specified on the purchase order, the insulators furnished under these specifications shall conform in all respects to the requirements hereinafter prescribed. The text and the drawings supplement each other and both shall be considered as parts of these specifications.

(b) The manufacturer shall submit

drawings of insulators of the class (Table I) covered in the inquiry or order. The drawings shall show the outline of the insulators, height, diameter, sizes of wire grooves, length of pin cavity, and relation of the dome of the pin cavity to the position of the wire grooves. Any variations in dimensions from those specified in Table I, due to the process of manufacture, shall be so indicated on the drawings.

### Material

3. (a) The insulators shall be of lime glass of the grade generally accepted for power insulators of this class, of good quality and homogeneity. The glass shall be either colorless or colored, as specified in the purchase order.

(b) The insulators shall be free from chips and cracks, and stones greater than  $\frac{1}{16}$  in. in diameter. All portions of the insulators shall be reasonably free from surface roughness, checks and unevenness, holes, stones, air bubbles and oil and gas stains. They shall in no case contain fins, mold seam marks, shear marks,

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> These specifications are identical in substance but not in form with the Specifications for Low and Medium Voltage Pin-Type Lime-Glass Insulators (TD-51—1942), approved by the Transmission and Distribution Committee, Edison Electric Institute.

molded projections, or sharp edges of such proportions as to scratch or injure the hands when being handled. The pin cavity shall be entirely free from surface checks. The wire groove or rounded edge of the wire groove shall contain no irregularity of such proportion as to abrade the conductor or its covering.

### Definitions

4. (a) *Chips and Spalls*.—Chips and

ing mold seam marks, or portions of the glass mass visible in the finished article extruded beyond the design contour, into spaces between mold parts, rings, or plungers.

(g) *Folds*.—Folds are linear indentations of the surface of the insulator caused by the meeting and partial welding of a re-entrant portion of a surface which has been partially chilled in some previous stage of the process of manufacture.

TABLE I.—INSULATOR CHARACTERISTICS.

	Class					
	No. 1 <sup>a</sup>	No. 1A <sup>b</sup>	No. 2	No. 3	No. 4	No. 5
Flashover (low frequency), kv. .... (Dry.....)	35	40	50	60	70	85
..... (Wet.....)	20	20	25	30	40	50
Critical impulse flashover, (Positive....)	50	65	80	95	115	140
(1½ by 40 microsecond wave), kv. (Negative....)	70	85	100	120	140	170
Leakage distance, min., in. ....	3¾	4	4½	6½	7½	11¼
Dry arcing distance, min., in. ....	2	2½	3	4	4½	6
Nominal pin hole diameter, c in. ....	1	1	1	1	1	1½
Length of thread, in. ....	1¾ (+¼/-0)	1¾ (+¼/-0)	1¾ (+¼/-0)	1¾ (+¼/-0)	2 ±¼	2 ±¼
Radius of side groove, in. ....	¾ ±½	¾ ±½	¾ ±½	¾ ±½	¾ ±½	¾ ±½
Radius of top groove, in. ....	¾ ±½	¾ ±½	¾ ±½	¾ ±½	¾ ±½	¾ ±½
Thickness of wall surrounding thread, min., in. ....	¼	¼	¼	¼	¼	¼
Mechanical strength, cantilever, lb. ....	2 500	2 500	2 500	2 500	2 500	2 000

<sup>a</sup> Class No. 1 covers insulators with side groove only.

<sup>b</sup> Class No. 1A covers insulators with large top and side grooves.

<sup>c</sup> If other size pin holes are desired, the purchaser shall so specify.

spalls are defects caused by the fracture and removal of a portion of the insulator.

(b) *Cracks*.—Cracks are free surfaces formed in the body of the glass by overstress.

(c) *Surface Checks*.—Surface checks are shallow cracks penetrating only slightly below the surface of the glass.

(d) *Stones*.—Stones are foreign solid inorganic matter completely or partially surrounded by glass usually consisting of particles of refractory material, crystalline products due to combination of refractory and glass batch constituents, particles of incompletely melted batch or products of devitrification of the glass.

(e) *Air Bubbles*.—Air bubbles are visible gaseous inclusions within the body of the glass.

(f) *Fins*.—Fins are outwardly extend-

TABLE II.—SIZE OF SAMPLE FOR INSPECTION AND TEST.

	Number of Insulators in Lot		
	0 to 5 000	5 001 to 10 000	Over 10 000
Inspection tests:			
Visual.....	32	46	60
Gaging.....	10	15	20
Mechanical tests:			
Thermal shock.....	10	15	20
Cantilever strength.....	3	4	5
Electrical tests:			
Dry flashover.....	3	4	5
Wet flashover.....	3	4	5
Impulse flashover.....	3	4	5
Total number of samples.....	32	46	60

(h) *Shear Marks*.—Shear marks are marks caused by the incomplete welding of a surface sheared during manufacture, or by the inclusion of foreign matter under such a surface.

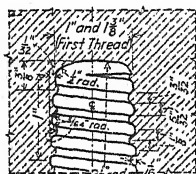
(i) *Oil and Gas Stains*.—Oil and gas

stains are smoky films caused by deposition from mold swabbing or products of combustion during annealing.

### Insulator Characteristics

5. (a) Insulators shall be furnished in classes having the characteristics specified in Table I.

(b) The pin cavity shall be centered in the insulator, its top shall be concave and the threads shall be in accordance with Fig. 1.



1" and  $\frac{1}{8}$ " Thread in Insulator

Note: Thread fit to be checked with insulator gage

NOTE.—Four threads per inch, tapering  $\frac{1}{8}$  in. in diameter to 1 in. in length.

FIG. 1.—Pin Cavity Dimensions.

(c) When subjected to the gaging test described in Section 12, the dome clearance of the insulator shall be not less than  $\frac{1}{4}$  nor more than  $\frac{5}{8}$  in. There shall be no perceptible play or rocking of the insulator when seated on the gage. The number of turns necessary to release the gage from the insulator shall be not less than 3.

### Marking

6. Insulators shall bear the manufacturer's symbol or identification mark, the catalog number, and the mold number. These designations shall be in raised characters, smoothly molded on the glass. The designations shall be free from sharp edges and projections and shall not extend more than  $\frac{3}{16}$  in. above the surface of the insulator. The height of the characters shall not exceed  $\frac{3}{16}$  in. The greatest dimension of the manufacturer's symbol or identification mark shall not exceed  $\frac{5}{8}$  in.

### Packing

7. (a) The insulators shall be so packed as to prevent injury to them during transportation and handling. Insulators packed in containers conforming to all construction requirements of Consolidated Freight Classification as to bursting tests, size limit, and gross weight shall be considered as being properly packed.

(b) Each box or carton for carload shipment shall be marked with the number and description of insulators, the catalog number, and the manufacturer's name and address.

(c) Each box or carton for L.C.L. (less carload) shipment shall be marked with the purchaser's name and shipping address, purchaser's order number, the number and description of insulators, the catalog number, the gross weight, and the manufacturer's name and address.

(d) In addition, boxes or cartons of any shipment may be marked as specified in Section 8 (d).

### Manufacturer's Production Sampling and Tests

8. (a) Every insulator shall be inspected during production for structural defects. At least one insulator out of each 50 shall be selected at random and gaged. At least one insulator shall be selected at random during every hour of production, but not less than one out of every 1000 insulators, and given thermal shock and cantilever strength tests. Tested insulators shall be discarded and all insulators gaged with a steel gage shall be discarded.

(b) Specimens representative of the product shall be selected and given electrical flashover tests. These tests shall be made and certified by a commercial or institutional testing laboratory or by the manufacturer.



(c) A record shall be kept by the manufacturer of tests made on each day's production of insulators and shall include the following:

(1) A description of the material, the name and type, date of manufacture, and number of insulators in the day's production, and

(2) The number of insulators tested and the results of each of the routine tests, including:

The hot to cold and cold to hot thermal shock sustained,

The minimum number of turns to release the gage and limits of clearance in inches between the top end of the gage and the crown of the insulator, and

The minimum and average value of the cantilever strength tests in pounds.

These records shall be available to the purchaser at his request.

(d) Insulators inspected and tested during production in accordance with the above methods may be so designated by imprinting on the container the date of manufacture, and the statement that they were tested in accordance with these specifications, together with a reference to the test record by number.

#### **Purchaser's Acceptance Sampling**

9. (a) The lot of insulators offered for inspection shall be so arranged as to facilitate the work of the inspector.

(b) Sample insulators from each mold shall be selected from the lot at random. The number selected shall be as specified in Table II.

#### **Purchaser's Acceptance Tests**

10. (a) The sample for the purchaser's acceptance test shall be selected in accordance with Section 9 (b) and the

size of sample shall be in accordance with Table II.

NOTE.—This section is divided into three parts: Inspection, Mechanical, and Electrical, in order that the purchaser may select and require any or all parts.

#### *Inspection Tests*

(b) *Design and Dimensions*.—At least one insulator from each mold shall be selected from the sample and compared with the drawing. Failure of any of these specimens to conform, within manufacturing tolerances, to the dimensions on the drawing shall be cause for rejection of all insulators made in that mold.

(c) *Visual*.—The sample shall be visually inspected in accordance with Section 11. Failure of more than one specimen to conform to the requirements prescribed in Section 3 (a) and (b) shall be cause for rejection of the entire lot.

(d) *Gaging*.—The sample shall be gaged in accordance with Section 12. Failure of over 5 per cent of the sample to conform to the requirements prescribed in Section 5 (c) shall be cause for rejection of the entire lot.

#### *Mechanical Tests*

(e) *Thermal Shock*.—The sample, containing no specimens which have been gaged, shall be subjected to the thermal shock test in accordance with Section 13. If over 10 per cent of the sample fails to meet the thermal shock test, a retest on double the number shall be made. If over 10 per cent of this retest fail, the entire lot shall be rejected.

(f) *Mechanical Strength*.—The sample, containing no specimens which have been gaged, shall be subjected to the mechanical strength test in accordance with Section 14. The average strength of the specimens tested shall not be less than that specified in Table I, and the minimum strength of any one insulator shall not



be less than 60 per cent of the average of the sample. Failure of the sample to conform to these requirements shall be cause for rejection of the entire lot.

### *Electrical Tests*

(g) *Certification*.—Unless otherwise specified in the inquiry or order, the purchaser, if so desired by the manufacturer, shall accept as evidence of the compliance of the insulator with the electrical tests, certificates of tests carried out on not less than three insulators identical in all essential details with those purchased.

Certificates of all these tests, together with a record of any alterations, whether essential or not, which have been made to the insulators since the tests were carried out, shall be held available by the manufacturer for inspection.

Electrical tests shall be conducted and certified by a commercial or institutional testing laboratory or by the manufacturer, unless the purchase order specifies otherwise.

Electrical tests made by other than the manufacturer shall be at the expense of the purchaser. Insulators for electrical tests shall be selected and provided by the purchaser.

(h) *Low-Frequency Dry and Wet Flashover*.—The sample shall be subjected to dry-flashover tests in accordance with Sections 8 and 9 of the Standard Methods of Testing Pin-Type, Lime-Glass Insulators (A.S.T.M. Designation: D 468),<sup>4</sup> and wet-flashover tests in accordance with Sections 10 and 11 of A.S.T.M. Methods D 468. Failure of any insulator to conform to the requirements prescribed in Table I shall be cause for rejection of the entire lot.

(i) *Impulse Flashover*.—The sample shall be subjected to impulse flashover tests in accordance with Sections 12 and

13 of A.S.T.M. Standard Methods D 468. Failure of any insulator to conform to the requirements prescribed in Table I shall be cause for rejection of the entire lot.

### METHODS OF TESTING

#### Visual Inspection

11. The visual inspection shall consist of checking for such physical and structural defects as prescribed in Section 3 (a) and (b).

#### Gaging

12. A gage of the design and dimensions shown in Fig. 1 of A.S.T.M. Methods D 468 shall be screwed tightly into the insulator pin cavity and the dome clearance (distance between line "A" of the gage drawing and the crown of the pin cavity) shall be measured. There shall be no perceptible play or rocking of the insulator when seated on the gage. The gage shall then be removed from the insulator, counting the number of turns to disengage. All insulators which have been gaged shall be discarded.

#### Thermal Shock Test

13. The specimen insulators shall be subjected to the stipulated thermal shock test (manual method or machine method) as described in Section 5 of A.S.T.M. Methods D 468. Tested insulators shall be discarded.

#### Mechanical Strength Test

14. The specimen insulators shall be subjected to the mechanical strength test as described in Section 7 of A.S.T.M. Methods D 468.

#### Electrical Voltage Tests

15. The specimen insulators shall be subjected to the specified dry-flashover, wet-flashover, and critical impulse-flashover tests as described in Sections 8 to 13 of A.S.T.M. Methods D 468.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# *Tentative Specifications for*

## COMMUNICATION AND SIGNAL PIN-TYPE LIME-GLASS INSULATORS<sup>1</sup>



A.S.T.M. Designation: D 879 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover pin-type lime-glass insulators for use in supporting conductors on open-wire communication and signal lines.

### Material

2. The insulators shall be of lime glass of the grade generally accepted for communication and signal line work, of good quality and homogeneity. The glass used shall be transparent, and if not colorless shall be only slightly tinted. Insulators shall be manufactured and processed in a careful and workmanlike manner, in accordance with good design and commercial practice.

### Thermal Shock

3. The glass insulators shall be capable of standing, without cracking, checking or chipping, hot-to-cold and cold-to-hot thermal shock tests as follows:

*Hot-to-Cold Test.*—The insulators

shall be immersed for 10 min. in a hot water bath (120 to 140 F.) and then transferred to a cold water bath and left therein for 10 min. The temperature difference between the hot and cold baths shall be  $70 \pm 2$  F.

*Cold-to-Hot Test.*—The insulators, after having been subjected to the hot-to-cold test described above, shall be immersed in a cold water bath after 10 min., and then transferred to a hot water bath and left for 10 min. The temperature difference between the cold and hot baths shall be  $100 \pm 2$  F.

### Electrical Characteristics

4. The electrical characteristics of the material when measured at 1000 cycles per second in accordance with Section 13 shall be such that the dielectric constant will be not more than 7.9, and the product of the power factor and the dielectric constant will be not more than 0.28.

### Dimensions and Permissible Variations

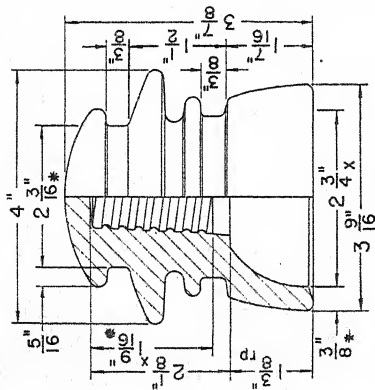
5. (a) *Design and Construction.*—Pin-type lime-glass insulators shall conform to the requirements for physical dimen-

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

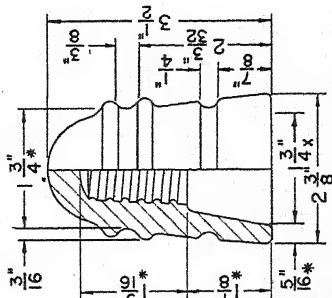
<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

Prior to their present publication as tentative, these specifications were published as Emergency Specifications ES-41 from 1944 to 1946, being revised in 1946 and issued as tentative.

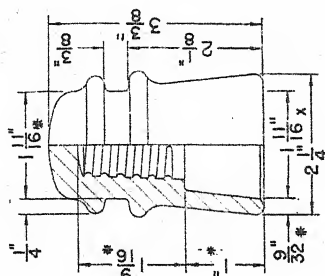




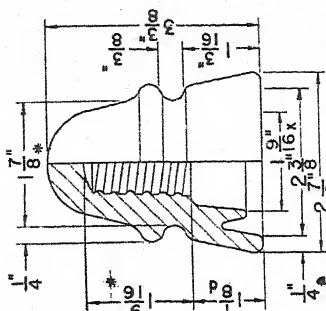
TW



# DOG-PONY



Y  
Z  
O  
D



DP-PONY

$\left\{ \begin{array}{l} \text{ce} = +\frac{1}{16} - \frac{1}{4}; \text{rp} = +0.0005 - \frac{5}{16}; \text{d} = \pm \frac{1}{8} \\ * = \text{MINIMUM}, \text{X} = \pm \frac{3}{16} \end{array} \right. \text{DIAMETER AND HEIGHT} \pm \frac{1}{8}$   
 DEPTH AND WIDTH OF WIRE GROOVE  $\pm \frac{1}{16}$

## STYLE SYMBOLS

CSC =CARRIER CURRENT INSULATOR FOR STEEL  
PINS CUSHIONED WITH THIMBLES

TOLL =STANDARD BELL SYSTEM TOLL LINE INSULATOR

EXCHANGE = STANDARD BELL SYSTEM EXCHANGE LINE  
INSULATOR

DP = DOUBLE PETTICOAT

DPG-DP = DEEP GROOVE DOUBLE PETTICOAT

TPG-WU = TOP GROOVE WESTERN UNION

TTW = ONE PIECE TRANSPOSITION

PONY = SINGLE GROOVE SINGLE PETTICOAT

DOG-PONY=DOUBLE GROOVE PONY

DP-PONY = DOUBLE PETTICOAT PONY

EACH OF THE ABOVE ILLUSTRATED INSULATORS IS INTENDED FOR SUPPORTING OPEN WIRES ON COMMUNICATION AND SIGNAL LINES. THE CSC INSULATOR IS DESIGNED FOR MOUNTING ON STEEL PINS CUSHIONED WITH .016 INCH LEAD THIMBLES OR AN APPROVED EQUIVALENT. THE OTHER INSULATORS SHOWN ARE DESIGNED FOR MOUNTING ON WOODEN INSULATOR PINS, WOODEN POLE BRACKETS OR STEEL INSULATOR PINS EQUIPPED WITH WOODEN THIMBLES.

FIG. 1,---Styles of Insulators.

sions in accordance with the applicable style shown in Fig. 1, and shall give evidence of satisfactory workmanship in accordance with the requirements prescribed in the following Paragraphs (b) to (e).

(b) *Screw Threads*.—The insulators shall have the standard screw threads to fit the wooden-pin gage or the CSC gage, as applicable. The dimensions and construction of these gages shall be as described in Section 6 and in Figs. 2 and 3 respectively of the Standard Methods of Testing Pin-Type, Lime-Glass Insulators (A.S.T.M. Designation: D 468).<sup>3</sup> The thread shall be smooth, of uniform pitch, and well-centered in the insulator.

(c) *Gage Fit*.—When the insulator gage is fully inserted in the insulator it shall conform to the following requirements:

(1) The end of the wooden pin gage shall be not more than  $\frac{3}{16}$  in. from the apex of the insulator pin cavity; the shoulder of the CSC gage shall bear firmly against the insulator.

(2) At least two and one-half revolutions shall be required to remove the insulator from the gage.

(3) When the gage has been unscrewed one complete turn, it shall not be possible to rock the gage into contact with the inner surface of the petticoat.

(d) *Diameter and Height*.—The diameter and height shall not vary more than plus or minus  $\frac{1}{8}$  in. from that shown on the applicable style in Fig. 1.

(e) *Depth and Width of Wire Groove*.—The depth and width of wire groove shall not vary more than plus or minus  $\frac{1}{16}$  in. from that shown on the applicable style in Fig. 1.

### Workmanship and Finish

6. (a) *Visible Defects*.—So far as practicable, the insulators shall have a finish insuring smooth even surfaces and free-

dom from cracks, checks, bubbles, sharp edges and other flaws, of a character detrimental to the strength or life of the insulator.

(b) *Drip Points*.—Insulators conforming in all other respects to the requirements of these specifications, but having on the lower edge of their outer petticoat a series of projecting drip points, may be accepted under these specifications provided these points are free from sharp or broken edges.

### Marking

7. The insulators shall bear, on the outside curved surface near the bottom, the name or brand of the manufacturer, a numeral to designate the mold used in their manufacture, and a number or listing, employed to designate the insulator. Such designations shall be in raised characters, smoothly molded in the glass; they shall be free from sharp edges or projections and shall not extend more than  $\frac{3}{32}$  in. above the surface of the insulator.

### Packaging

8. The insulators shall be packaged in the quantities listed in Table I within

TABLE I.—PACKING AND WEIGHTS.

Style	Units per Carton	Cartons Arrangement of Insulator, Length by Width by Depth	Shipping Containers		
			Approximate Net Weight, lb.	Packed Cartons per Shipping Container	Approximate Gross Weight, lb.
CSC.....	50	5 by 5 by 2 in.	56	3	200
DP.....	40	5 by 4 by 2 in.	65	2	160
Toll.....	50	5 by 5 by 2 in.	54	3	207
TW.....	30	5 by 3 by 2 in.	58	3	210
Exchange.....	50	5 by 5 by 2 in.	36	4	210
TpG-WU.....	40	5 by 4 by 2 in.	72	2	165
DpG-DP.....	50	5 by 5 by 2 in.	58	3	195
Pony.....	50	5 by 5 by 2 in.	31	4	158
DoG-Pony.....	50	5 by 5 by 2 in.	32	4	158
DP-Pony.....	50	5 by 5 by 2 in.	41	4	195

a regular slotted corrugated fiberboard carton. The insulators shall be placed in the carton within the cells of inter-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

locking partitions fabricated of 200-lb. Mullen test solid or corrugated fiberboard. Each partition cell shall contain one insulator. The insulators shall be packed within the carton in two layers or tiers with the layers separated by a fiberboard pad and the space between the inner flaps of the container (top and bottom) shall be filled with a fiberboard pad, the pads to be fabricated of material similar to the interlocking partitions. Insulators in the top layer shall be in the same relative position as the ones directly below; that is, top to bottom or bottom to top.

NOTE.—For packaging for the Armed Forces reference should be made to the Army-Navy General Specification for Packaging and Packing for Overseas Shipment (U. S. Army Specification No. 100-14A; U. S. Navy Specification 39 P 16a).

## METHODS OF TESTING

### *Qualification Approval Tests*

#### Qualification Approval Tests

9. (a) For qualification, tests shall be made at a designated laboratory on a sample quantity to insure that the basic design produces insulators conforming to all the requirements of these specifications. Qualification tests shall be conducted in the order in which they are listed in Table II.

TABLE II.—OUTLINE OF QUALIFICATION TESTS.

Test	Number of Specimens	Failures Permitted
Thermal shock.....	48	4
Visual inspection and physical measurement.....	48	0
Electrical (made on material plaques)...	6	0

(b) If any specimen fails the visual inspection and physical measurement test or the electrical test, or if more than 4 specimens fail the thermal shock test, as prescribed in Sections 11 to 13, the insulator manufacturer may make application to submit a second lot of specimens for test.

## Test Specimens

10. Forty-eight specimens of each style of insulator for which qualification approval is desired shall be submitted for qualification tests. Manufacturers' test readings for all tests not damaging to the insulators for at least one unit of each style submitted, shall accompany the specimens. This information shall include identification of the test equipment used in making the tests. A minimum of six plaques of the same material as the insulator specimens shall be furnished, for the style of insulator submitted or for the group of styles, if more than one style is submitted at one time, for electrical tests.

## Thermal Shock Test

11. The specimen insulators shall be subjected to the stipulated thermal shock test (manual method or machine method) as described in Section 5 of A.S.T.M. Methods D 468.

## Visual Inspection and Measurement of Dimensions

12. The insulators shall be inspected and gaged to verify that they conform to the requirements prescribed in Sections 5 and 17.

## Electrical Tests

13. Dielectric constant and power factor tests shall be made on six test specimens in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

## *Inspection Tests*

### Inspection Tests

14. (a) Each insulator offered for delivery shall be subjected at the place of manufacture to visual inspection and physical measurements in accordance with Sections 5 (a) and 17, except that

only every fiftieth insulator shall be gaged in accordance with Section 5 (c).

(b) Production-sampling tests shall be made on a relatively small number of insulators taken from production and, in general, are intended to check production quality with reasonable thoroughness.

#### Test Equipment and Inspection Facilities

15. The insulator manufacturer shall furnish and maintain all necessary facilities and equipment for making all inspection tests, and shall perform all tests under the supervision of the inspector. The test equipment shall be adequate in quantity to enable inspection to keep up with production. The test equipment shall be of sufficient accuracy and quality to permit performance of the required tests. Unless otherwise specified, inspection of insulators shall be made at the place of manufacture.

#### Test Specimens

16. (a) For production sampling, insulators shall be selected by the inspector, from a continuous production run of insulators that have passed the production tests. The number of specimens used for the various tests shall be in accordance with Table III, unless otherwise specified in the order.

TABLE III.—NUMBER OF SPECIMENS FOR PRODUCTION-SAMPLING TEST AND FAILURES PERMITTED.

Test	Quantity in Each Style in Daily Production					
	0 to 5000		5001 to 10 000		Over 10 000	
	Number of Specimens	Failures Permitted	Number of Specimens	Failures Permitted	Number of Specimens	Failures Permitted
Visual inspection and physical measurement.....	40	0	60	0	80	0
Gaging.....	20	0 <sup>a</sup>	30	0 <sup>a</sup>	40	0 <sup>a</sup>
Thermal shock.....	20	2	30	3	40	4

<sup>a</sup> See Section 17 (c).

(b) Each insulator in the sample lot shall be subjected to visual inspection in

accordance with Section 17. The sample lot shall then be divided into two groups. One group shall be subjected to the test outlined in Section 17(c). The other group shall be subjected to the tests outlined in Section 18.

#### Visual Inspection and Measurement of Dimensions

17. (a) *Visual Inspection*.—All of the insulators in the production-sampling lot shall be inspected for surface defects in accordance with Section 6. Failure of any insulator under this test shall be cause for reinspection of the entire production lot for this requirement.

(b) *Measurement of Dimensions*.—All of the insulators in the production-sampling lot shall be compared with the drawing. Failure of any of the specimens to conform within the manufacturing tolerances to the dimensions of the drawing shall be cause for rejection of the production lot.

(c) *Gaging*.—One half of the sample lot of insulators shall be gaged in accordance with Section 5. Failure of more than one insulator under this test shall be cause for rejection of the production lot. If only one insulator fails, a second selection shall be made and shall include at least five insulators having the same mold number as the insulator that failed. Failure of any one insulator in the second lot shall be cause for rejection of the production lot.

#### Thermal Shock Test

18. The other half of the sample lot shall be tested for thermal shock in accordance with A.S.T.M. Methods D 468. If more than 10 per cent of the sample lot fails this test, a retest shall be made on a new sample lot of insulators of double the number previously tested, selected from the same production lot. Failure of more than 10 per cent of the second sample lot shall be cause for rejection of the production lot.



# Tentative Methods of Test for POWER FACTOR AND DIELECTRIC CONSTANT OF ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 150 - 46 T

ISSUED, 1922; REVISED, 1923, 1927, 1931, 1932, 1934, 1935, 1936, 1939, 1941, 1942, 1944, 1945, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods provide procedures for the determination of the power factor and dielectric constant of solid and fluid electrical insulating materials at frequencies from 25 cycles per second to 100 megacycles per second. These procedures include five bridge arrangements, and three resonant-circuit methods. The following tabulation gives ranges of frequencies for which the various procedures are generally deemed applicable; these ranges overlap to a considerable extent and the suitability indicated for one procedure is therefore not to be interpreted as excluding other procedures at a given frequency:

Method	Reference		Usual Frequency Range, cycles per second
	Section	Figure	
Conjugate Schering Bridge.....	22	7	25 to 5 000 000
High-Voltage Schering Bridge.....	23	8	25 to 10 000
Parallel Resistance Bridge.....	24	9	1 000 to 100 000
Series Resistance Bridge.....	25	10	200 to 100 000
Transformer Bridge.....	26	11-12	25 to 1 000
Resonant Circuit, Resistance Variation...	27	13	100 000 to 1 000 000
Resonant Circuit, Susceptance Variation...	28	14	10 000 to 100 000 000
Resonant Circuit, Resonance Rise.....	29	15	50 000 to 10 000 000

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

## Definitions

2. (a) *Dielectric Constant*.—The dielectric constant of a dielectric material is that property which determines the electrostatic energy stored in the material per unit volume per unit potential gradient. For the purposes of these methods the dielectric constant is the ratio of the equivalent parallel capacitance of a capacitor in which the material is the dielectric, measured at the specified frequency, to the capacitance of the same capacitor with a vacuum as the dielectric, and is represented by the symbol  $K$ .

NOTE.—The dielectric constant of air under normal conditions may be considered equal to that of a vacuum.

(b) *Dielectric Phase Angle*.—The dielectric phase angle is the angular difference in phase between the sinusoidal voltage applied to the dielectric and the component of the resulting current which has the same frequency as the applied voltage.

(c) *Dielectric Loss Angle*.<sup>3</sup>—The dielectric loss angle is the difference be-

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> The terms "dielectric phase defect angle" and "dielectric phase difference," with others, have been used in technical literature for this quantity.



tween ninety electrical degrees (90 deg.) and the dielectric phase angle.

(d) *Dielectric Dissipation Factor*.—The dielectric dissipation factor of a material is the cotangent of the dielectric phase angle or the tangent of the dielectric loss angle, and is represented by the symbol  $D$ .

NOTE.—The storage factor or quality factor,  $Q$ , is the reciprocal of the dissipation factor,  $D$ .

(e) *Dielectric Power Factor*.—The dielectric power factor of a material is the cosine of the dielectric phase angle or the sine of the dielectric loss angle.

NOTE.—When the cotangent of the phase angle is smaller than 0.1, the cosine and the cotangent differ by less than 0.0005 and the power factor may be considered equal to the cotangent of the phase angle or the tangent of the loss angle. When the measured cotangent proves to be larger than 0.1, the correction to be applied to the observed value to secure the power factor may be obtained from the curve of Appendix I, or the corresponding cosine may be taken from trigonometric tables.

(f) *Dielectric Loss Factor*.—The dielectric loss factor of a material is the product of its dielectric constant and its dissipation factor.

## THEORY OF TEST

### Parallel Notation

3. Any insulating material between two electrodes constitutes a capacitor. Such a physical capacitor may be represented by a "pure" capacitance,  $C_p$

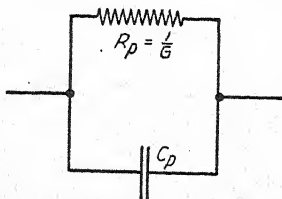


FIG. 1.—Equivalent Parallel Circuit.

(loss angle = 0), and a resistance,  $R_p$ , in parallel, as shown in Fig. 1. The resistance,  $R_p$ , is called the equivalent

parallel resistance and the capacitance,  $C_p$ , is called the equivalent parallel capacitance. For sine-wave voltage and current the vector relations are as shown in Fig. 2. For this notation the dissipa-

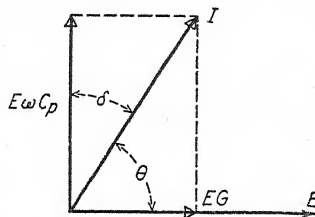


FIG. 2.—Vector Diagram for Equivalent Parallel Circuit.

tion factor and the power factor are as follows:

$$D = \cot \theta = \tan \delta = \frac{1}{\omega C_p R_p} = \frac{G}{\omega C_p}$$

Power factor =  $\cos \theta = \sin \delta = \sin \tan^{-1} D$ , where:

$C_p$  = capacitance in farads,

$R_p$  = resistance in ohms,

$G$  = conductance in mhos, and

$\omega = 2 \pi$  times the frequency in cycles per second.

As explained in the Note under Section 2 (e), when  $\tan \delta$  is less than 0.1, power factor and dissipation factor differ by less than 0.0005.

### Series Notation

4. A physical capacitor may also be represented by a "pure" capacitance,  $C_s$ , and a resistance,  $R_s$ , in series as shown in Fig. 3. The resistance,  $R_s$ , is called

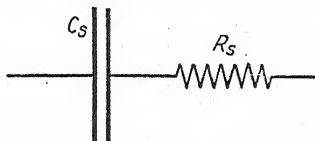


FIG. 3.—Equivalent Series Circuit.

the equivalent series resistance and the capacitance,  $C_s$ , is called the equivalent series capacitance of the capacitor. For sine-wave voltage and current, the vector relations are as shown in Fig. 4. The

dissipation factor and the power factor are as follows:

$$D = \cot \theta = \tan \delta = \omega C_s R_s$$

$$\text{Power factor} = \cos \theta = \sin \delta = \sin \tan^{-1} D$$

where:

$C_s$  = capacitance in farads,

$R_s$  = resistance in ohms, and

$\omega = 2 \pi$  times the frequency in cycles per second.

As explained in the Note under Section 2 (e), when  $\tan \delta$  is less than 0.1, power

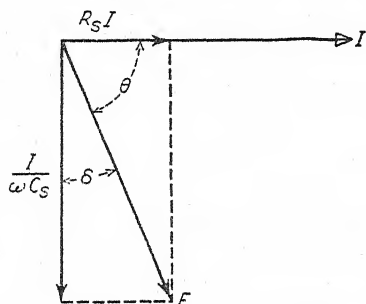


FIG. 4.—Vector Diagram for Equivalent Series Circuit.

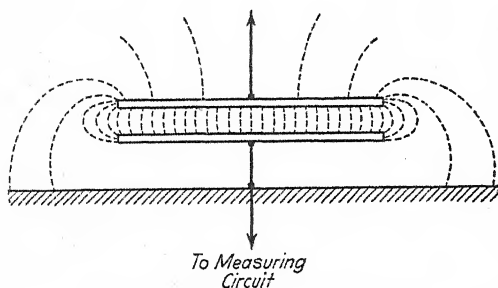


FIG. 5.—Simple Capacitor Circuit.

factor and dissipation factor differ by less than 0.0005. Under these conditions:

$$\text{Power factor} = D = \omega C_s R_s$$

### Equivalency of Notation

5. The equivalent parallel capacitance and the equivalent series capacitance of a given physical capacitor are not identical, the difference being dependent

on the loss angle. If capacitance has been determined as a series capacitor, the equivalent parallel capacitance may be calculated from the following relation:

$$C_p = \frac{C_s}{1 + D^2} = \frac{C_s}{1 + \tan^2 \delta} = C_s \cos^2 \delta$$

The two capacitances are alike within 1.0 per cent unless  $D$  exceeds 0.10; within 0.1 per cent unless  $D$  exceeds 0.03.

### Dielectric Constant

6. (a) With a capacitor formed as shown in Fig. 5, with only the opposing

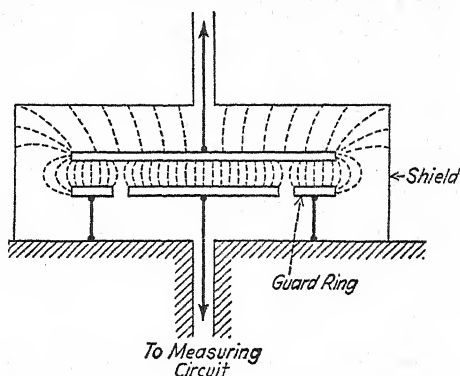


FIG. 6.—Capacitor With Guard Ring and Shield.

surfaces of the two metallic electrodes separated by the dielectric material under test, the total capacitance,  $C_s$ , measured between the electrodes will be the sum of the following:

$C_p$ , the “equivalent parallel capacitance” (Section 3), represented in Fig. 5 by the parallel flux lines in the dielectric material between the electrodes,

$C_e$ , the edge correction, represented by the curving flux lines in air, from one electrode to the other, and

$C_o$ , the stray capacitance, represented by the flux lines in air passing from the “high” electrode to space and to earth.

(b) When the dielectric material under

test is replaced by air (vacuum), the electrodes' spacing and alignment remaining unchanged, and with  $C_s$  and  $C_e$  remaining presumably unaltered, the equivalent parallel capacitance,  $C_p$ , will become the "interelectrode capacitance,"  $C_a$ ; and by the definition in Section 2 (a), the dielectric constant,  $K$ , is given by the following equation:

$$K = \frac{C_p}{C_a} = \frac{C_s - C_e - C_g}{C_a}$$

(c) Use of a guard ring (see Fig. 6) makes it possible to exclude the edge correction,  $C_e$ , from the measured capacitance,  $C_s$ ; and a shield about the guarded electrode (and usually the "high" electrode also) can be used to eliminate the stray capacitance,  $C_g$ , from the measured capacitance. (See Section 21 on Shielding.) Under these conditions, attainable in the bridges described in Sections 22 to 26,  $C_s$  becomes simply  $C_p$ , and

$$K = \frac{C_s}{C_a} = \frac{C_p}{C_a}$$

#### Formulas for Interelectrode Capacitance, $C_a$

7. The following formulas are useful for the calculation of the interelectrode capacitance,  $C_a$ , in micro-microfarads with air (vacuum) as the dielectric: where:

- $A$  = area of flat plates,
- $d$  = diameter of flat circular plates,
- $d_1$  = inside diameter of tube of dielectric material,
- $d_2$  = outside diameter of tube of dielectric material,
- $d_m = \frac{d_1 + d_2}{2}$  = average diameter of tube,
- $t$  = thickness of sheet, layer, film, or tube-wall of dielectric material,
- $L$  = length of the cylindrical electrode, and

$\log$  = logarithm of a number to the base 10 (Briggs' logarithm).

(a) *For flat opposing parallel plates:*

$$C_a = 0.08854 \frac{A}{t}, \text{ when dimensions are in cm.}$$

$$C_a = 0.2249 \frac{A}{t}, \text{ when dimensions are in in.}$$

(b) *For flat opposing parallel circular plates:*

$$C_a = 0.06954 \frac{d^2}{t}, \text{ when dimensions are in cm.}$$

$$C_a = 0.1766 \frac{d^2}{t}, \text{ when dimensions are in in.}$$

(c) *For coaxial cylindrical electrodes:*

$$C_a = \frac{0.2416 L}{\log \left( \frac{d_2}{d_1} \right)}, \text{ when dimensions are in cm.}$$

$$C_a = \frac{0.6137 L}{\log \left( \frac{d_2}{d_1} \right)}, \text{ when dimensions are in in.}$$

(d) *For coaxial cylindrical electrodes:<sup>a</sup>*

$$C_a = 0.2782 L \left[ \frac{d_m}{t} \right] \left( 1 - \frac{1}{3} \left[ \frac{t}{d_m} \right]^2 \right),$$

when dimensions are in cm.

$$C_a = 0.7065 L \left[ \frac{d_m}{t} \right] \left( 1 - \frac{1}{3} \left[ \frac{t}{d_m} \right]^2 \right),$$

when dimensions are in in.

<sup>a</sup> These approximate formulas are accurate to 0.1 per cent unless  $t/d_m$  is greater than  $\frac{1}{3}$ .

(e) *Guards:*

When a guard ring is used with a circular or a rectangular plate, or guard tubes with a cylindrical electrode, the effective area of the electrode is increased by one-half the area of the space between the guard and the electrode. Thus for circular plates, the  $d$  of Paragraph (b), should be replaced by  $(d + w)$ , and the  $L$  of Paragraphs (c) and (d) by  $(L + w)$ , where  $w$  = the width of gap between electrode and guard.

The width of a guard ring or the lengths of the guard tubes should be not less than five times the thickness of the dielectric.

#### Edge Correction, $C_e$ <sup>4</sup>

8. The edge correction per unit of length along the edge of the electrode is the same for circular, rectangular,<sup>5</sup> and cylindrical electrodes, but differs according to the various electrode arrangements. The following expressions apply to *thin electrodes* such as tin foil when the area of each of the electrodes is greater than 45 cm.<sup>2</sup> (7 in.<sup>2</sup>). When these expressions are used in the formula for  $K$  in Section 6 (b), this formula will be in error by less than 1 per cent. When electrodes are of unequal size, the inter-electrode capacitance,  $C_e$ , shall be calculated for the smaller electrode.

(a) *Electrodes Extending to Edge of Plate.*—The edge correction,  $C_e$ , expressed in micro-microfarads, for electrodes extending to the edge of a plate specimen is given by the following formulas if care is taken to see that the specimen has smooth sharp edges and that the electrodes come exactly to the edge:

$$C_e = \left(0.058 \log \frac{1.5}{t} + 0.0185\right) P, \text{ when dimensions are in cm.}$$

$$C_e = \left(0.147 \log \frac{0.59}{t} + 0.047\right) P, \text{ when dimensions are in in.}$$

where:

$P$  = distance around perimeter of the electrode (for example,  $\pi$  times the diameter for circular electrodes).

(b) *Equal Electrodes Smaller than Plate.*—In this case the edge correction is a function of  $K$ , the dielectric constant of the specimen. The edge correction,  $C_e$ , expressed in micro-microfarads, for thin electrodes of equal size and which are of the same shape as, but smaller than, the plate, is given by the following expressions, when proper care is taken to see that the electrodes are exactly opposite each other.

$$C_e = \left(0.058 \log \frac{1.5}{t} + 0.0185 K\right) P, \text{ when dimensions are in cm.}$$

$$C_e = \left(0.147 \log \frac{0.59}{t} + 0.047 K\right) P, \text{ when dimensions are in in.}$$

where:

$P$  = distance around perimeter of the electrode (for example,  $\pi$  times the diameter for circular electrodes).

The value of  $K$  is most easily found by considering  $C_e$  to be of the form:  $C_e = (M + NK)P$

Then:

$$K = \frac{C_x - MP - C_e}{C_e + NP}$$

(See the example given in Section 9.)

(c) *Unequal Electrodes on Plate.*—In this case also the edge correction is a function of  $K$ , the dielectric constant of the specimen. When thin electrodes are used, one of which extends at least five times the thickness of the plate beyond the edge of the other electrode, the edge correction,  $C_e$ , expressed in micro-microfarads is given by the following expressions:

<sup>4</sup> See extended treatment of this subject in papers by Harvey L. Curtis and Arnold H. Scott, "Edge Correction in Determination of Dielectric Constant," *Proceedings, Am. Soc. Testing Mats.*, Vol. 36, Part II, p. 815 (1936); Arnold H. Scott, "Determination of the Edge Correction in the Measurement of Dielectric Constant," *Proceedings, Am. Soc. Testing Mats.*, Vol. 37, Part II, p. 655 (1937); and Arnold H. Scott and Harvey L. Curtis, "Edge Correction in the Determination of Dielectric Constant," *Journal of Research, Nat. Bur. Standards*, Vol. 22, No. 6, p. 747 (1939). The last of the three references gives a more complete and more accurate treatment of edge correction than the other articles and includes formulas for a number of electrode arrangements in addition to those presented in these test methods.

<sup>5</sup> For rectangular electrodes the width of the electrode should be at least ten times the thickness of the specimen.

$C_e = \left(0.077 \log \frac{3.8}{t} + 0.0405 K\right) P$ , when dimensions are in cm.

$C_e = \left(0.195 \log \frac{1.5}{t} + 0.103 K\right) P$ , when dimensions are in in.

where:

$P$  = distance around the perimeter of the smaller electrode.

The value of  $K$  is most easily found by considering  $C_e$  to be of the form:

$$C_e = (M + NK)P$$

Then:

$$K = \frac{C_e - MP - C_g}{C_e + NP}$$

(See the example given in Section 9.)

(d) *Cylindrical Electrodes*.—When the ratio  $t/d_m$  is less than  $\frac{1}{10}$ , the edge correction  $C_e$  may be calculated from the equations given in Paragraphs (a) to (c) for the plate specimens, with  $P$  replaced by  $2\pi d_m$ . Analogous electrode arrangements may be used, namely, electrodes extending to the end of the tube, equal electrodes which are shorter than the tube and unequal electrodes where one electrode extends at least five times the thickness of the wall of the tube beyond the ends of the other cylindrical electrode.

When the ratio  $t/d_m$  is less than  $\frac{1}{8}$  and when the tube specimen extends beyond the electrodes at all times by at least five times the wall thickness, the edge correction may be eliminated from the determination by making two measurements of capacitance of the test specimen using a different length for the electrodes in the two cases. Care must be exercised in each of the measurements to keep the two electrodes of equal lengths and accurately opposite each other. For maximum accuracy of determination of the dielectric constant,  $L'$  should be about one-half of  $L$ . If  $C_p$  and  $C'_p$  are the measured capacitances corresponding to the lengths  $L$  and  $L'$  of

the cylindrical electrodes, the following expressions will give a value of dielectric constant with an error smaller than 1 per cent. (See also Section 7 (c) and (d)).

$$K = 3.60 \frac{(C_p - C'_p)t}{d_m(L - L')}, \text{ when dimensions are in cm.}$$

$$K = 1.415 \frac{(C_p - C'_p)t}{d_m(L - L')}, \text{ when dimensions are in in.}$$

When the method of measurement permits the use of a guard sleeve, the edge correction may be eliminated.

(e) *Thick Circular Electrodes*.—It is sometimes necessary to use for electrodes thick metal plates, or mercury pools of appreciable depth. The following formula<sup>6</sup> applies to equal circular electrodes, with sharp edges, extending to the edges of the circular specimen.

$$C_e = 0.0325 P \left( \log \frac{8P}{t} + Z - 1.305 \right), \text{ when dimensions are in cm.}$$

$$C_e = 0.0875 P \left( \log \frac{8P}{t} + Z - 1.305 \right), \text{ when dimensions are in in.}$$

where:

$P$  = perimeter of electrode,

$t$  = thickness of dielectric,

$b$  = thickness of each electrode,

$Z = (1 + X) \log (1 + X) - X \log X$ , and

$X = b/t$ .

When thick electrodes are used for routine testing a curve for the  $Z$  function may be plotted.<sup>7</sup>

**Stray Capacitance,  $C_g$**

9. The stray capacitance,  $C_g$ , may be eliminated from the capacitance measure-

<sup>6</sup>First published by G. Kirchhoff, Berlin (March, 1877). The reader should note that the symbols  $b$  and  $t$  have interchanged significance in the reference from that used in these methods.

<sup>7</sup>Such a curve is shown on p. 751 of the *Journal of Research, Nat. Bur. Standards*, Vol. 22 (1939), referred to in footnote 4.

ment by using the screened bridge methods shown in Figs. 7, 8, 9, 10, and 12. In the resonant circuit resistance variation method (Section 27) the electrostatic flux from the ungrounded plate to surrounding objects represents an additional capacitance which is effective in influencing the frequency of oscillation and must, therefore, be considered. The following equations giving the stray capacitance,  $C_s$ , expressed in micro-microfarads, for circular electrodes at the center of an infinite sphere may be used to calculate the approximate value of  $C_s$  if the specimen is at a distance of 30 cm. (12 in.) or more from surrounding objects:

$$C_s = 0.177 d, \text{ when dimensions are in cm.}$$

$$C_s = 0.449 d, \text{ when dimensions are in in.}$$

where:

$C_s$  = capacitance to earth (or space) of the ungrounded electrode, and

$d$  = diameter of the circular ungrounded electrode.

*Note: Example.*—Suppose a capacitor to be formed with tin foil electrodes, one of which is smaller than the other, and suppose its capacitance to be measured by the resonant-circuit substitution method:

Diameter of small electrode,	$d = 5.0$ in.
Thickness of specimen,	$t = 0.375$ in.
Measured Capacitance,	$C_x = 55.00 \mu\mu\text{f.}$
Stray Capacitance,	$C_s = 2.25 \mu\mu\text{f.}$
Inter-electrode Capacitance,	$C_a = 11.78 \mu\mu\text{f.}$

$$MP = 0.195 \log \frac{1.5}{t} P = 1.85 \mu\mu\text{f.}$$

$$NP = 0.103 P = 1.62 \mu\mu\text{f.}$$

$$K = \frac{C_x - MP - C_s}{C_a + NP} = 3.80$$

### Dielectric Loss Factor

10. The energy loss per unit volume per cycle per squared voltage gradient in an insulating material is equal to the product of its dissipation factor and its dielectric constant, which product, in turn, is dielectric loss factor.

$$\text{Loss factor} = D \times K$$

### TEST SPECIMENS

#### Test Specimens

11. Methods of test for a number of kinds of insulating materials have been standardized by the American Society for Testing Materials. These A.S.T.M. Standards should be consulted for special requirements as to test specimens, such as preparation, size, treatment, etc.

*NOTE.*—The following specifications and methods issued by the American Society for Testing Materials now contain special requirements covering power factor tests in addition to those described in these methods:

Tentative Specifications for Phenolic Laminated Sheet for Radio Applications (A.S.T.M. Designation: D 467),<sup>8</sup>

Tentative Specifications for Round Phenolic Laminated Tubing for Radio Applications (A.S.T.M. Designation: D 616),<sup>8</sup>

Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48),<sup>8</sup>

Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (A.S.T.M. Designation: D 176),<sup>8</sup>

Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295),<sup>8</sup> and

Standard Methods of Testing, Grading, and Classifying Natural Mica (A.S.T.M. Designation: D 351).<sup>8</sup>

(a) *Sheets and Plates.*—For testing sheet materials and molded or ceramic materials in plate form at frequencies up to 5 megacycles per second, the test specimen shall be, preferably, either a disk 4 to 6 in. (10.16 to 15.24 cm.) in diameter, or a square plate 4 to 6 in. (10.16 to 15.24 cm.) on a side. Preferably, the thickness shall be not less than 0.1 in. (0.254 cm.) nor more than 0.3 in. (0.762 cm.). Other thicknesses may be used where it is desired to test materials of the various thicknesses in commercial use. In any case, the capacitance of the specimen shall be, preferably, not less than 70  $\mu\mu\text{f.}$  However, it is not intended that this minimum value be rigidly adhered to,

<sup>8</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

provided the precision of the measurement meets the intended accuracy of the test method. When using the resonant circuit, resistance variation method, stray capacitances become troublesome and influence adversely the precision of measurement when the capacitance of the specimen is much less than  $70 \mu\mu\text{f}$ .

For tests made at frequencies from 1.0 to 100 megacycles per second, the test specimen shall be, preferably, in the form of a disk 2 in. (5.08 cm.) in diameter. However, the specimen diameter should be equal to, but may be less than, the diameter (see Section 28 (j)) of the electrodes of the capacitor which holds the specimen. Material in commercial use will determine the thickness of the specimen under test. In any case, the capacitance of the specimen shall be, preferably, not less than  $15 \mu\mu\text{f}$ . nor more than  $45 \mu\mu\text{f}$ . although satisfactory results may be obtained with specimen capacitances from 3 to  $80 \mu\mu\text{f}$ .

For all tests, the thickness of the specimen shall be uniform within plus or minus 5 per cent of the nominal thickness and the surface shall be as smooth and free as possible from irregularities.

(b) *Films*.—For testing films of insulating varnish, lacquer, or paints, the test specimen shall be prepared as specified for the dielectric strength tests on copper base in Section 11 of the Tentative Methods of Testing Varnishes Used for Electrical Insulation (A.S.T.M. Designation: D 115) of the American Society for Testing Materials.<sup>8</sup> The copper or brass plate shall be one capacitor plate, and tin foil the other. The capacitance of the specimen shall be, preferably, not less than  $70 \mu\mu\text{f}$ . However, it is not intended that this minimum value be rigidly adhered to, provided the precision of the measurement meets the intended accuracy of the test method. Both varnish films may be tested individually.

(c) *Tubes*.—For testing rolled or molded laminated tubes or ceramic tubes, the test specimen shall have the diameter and wall thickness of the material it represents, and shall be of sufficient length to provide a capacitance preferably not less than  $70 \mu\mu\text{f}$ . However, it is not intended that this minimum value be rigidly adhered to, provided the precision of the measurement meets the intended accuracy of the test method. The thickness shall be uniform to plus or minus 5 per cent, and the surfaces shall be smooth and free from irregularities.

(d) *Liquids*.<sup>8a</sup>—The liquid shall be tested in a suitable test cell in which the ratio of surface to thickness of liquid layer is sufficient to provide a capacitance preferably not less than  $70 \mu\mu\text{f}$ . However, it is not intended that this minimum value be rigidly adhered to, provided the precision of the measurement meets the intended accuracy of the test method. Three forms of test cells that have been found satisfactory for testing oils, filling and treating compounds, and other insulating liquids are shown in Appendix III. Cells of different constructions from those described may yield equally satisfactory results if the principles involved are scrupulously observed.

### Conditioning

12. The dielectric properties of most insulating materials are affected by temperature, humidity, pressure, exposure to air, etc. Hence, considerable importance may attach not only to the condition of the material at the time of measurement but also to the treatment it has undergone in the immediate past. Reference should be made to any standard for the material under test to learn what conditioning treatment has been specified. If such

<sup>8a</sup> Editorially revised in January, 1947.



specifications have not been formulated, the conditioning treatment to be given prior to measurement shall be agreed upon by the interested parties. Air temperatures in conditioning chambers with volumes of, for example, 4 cu. m. (140 cu. ft.) may readily be maintained constant within plus or minus 1 C. (1.8 F.). A satisfactory method of maintaining constant humidity in a chamber is given in Section 11 of the Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257) of the American Society for Testing Materials.<sup>8</sup> Whenever feasible, specimens should remain in the conditioning chamber during the measurements; the test leads must be suitably shielded (see Section 21).

## ELECTRODES

### Electrodes

13. (a) *Sheets and Plates*.—When the method of measurement will permit, a guard ring shall be employed. In this case the unguarded electrode shall be a foil disk of the same diameter as the specimen while the guarded electrode shall have a diameter equal to about 75 per cent of the specimen diameter with a guard ring extending to the edge of the specimen and leaving as narrow a space between guard ring and guarded electrode as is mechanically feasible. But in any case the radial width of the guard ring shall not be less than five times the thickness of the specimen. A very thin coating of a highly refined petrolatum or heavy mineral oil shall be used as an adhesive for foil electrodes. Such electrodes shall be applied under a smoothing pressure sufficient to eliminate all wrinkles. One very effective method is to use a narrow roller and to roll outward on the surface until no visible imprint can be made on the foil with the roller. The electrodes shall be ac-

curately centered with respect to each other and at least approximately centered with respect to the specimen. When foil electrodes are not used, it will be necessary to press the rigid metal electrodes against the specimen with a pressure of several hundred pounds per square inch, unless all surfaces are exactly plane and parallel. With materials of high dielectric constant (above 20), foil electrodes do not make sufficiently good contact and high-conductivity silver paint or sprayed metal must be used. Such electrode material is also satisfactory for specimens with low dielectric constant.

Mercury pool electrodes and guard ring may be employed but great care shall be exercised to prevent inclusion of small air bubbles between the mercury pool and the under surface of the specimen floating on the pool.

Electrode arrangements which have electrodes extending to the edge of the specimen are not the most precise because of the difficulty of preparing the specimens with good sharp edges and fixing the electrodes so that they extend exactly to the edges. The most accurate method for the determination of the dielectric constant, aside from the guard-ring method, is that using one electrode appreciably smaller than the other since then the electrodes do not have to be accurately centered on the specimen.

For measurement at frequencies from 0.1 to 100 megacycles per second by the susceptance variation method (Section 28), the electrodes must cover the full area of both sides of the test specimen. One satisfactory method of applying such electrodes is to start with foil at least as large as the specimen and trim it to size with a razor blade after it has been rolled on.

(b) *Films*.—For films less than about 0.05 in. in thickness the error in dielectric constant incurred by omitting the edge



correction will usually be less than 5 per cent. For still thinner films it will be necessary to take the same precautions as those recommended in Paragraph (a) for high dielectric constant materials. A guard ring, constructed as specified in Paragraph (a), shall be used when feasible.

(c) *Tubes*.—The outer electrode shall consist of tin foil or lead foil. The inner electrode shall consist either of tin foil or lead foil, or of mercury. When guard rings cannot be employed and the edge correction is to be eliminated by measurements with two lengths of electrodes, the electrodes shall be of the same length, accurately centered with respect to each other, and approximately centered with respect to the specimen. The tube shall extend beyond the larger electrodes by at least five times the wall thickness; the electrodes need be reduced in length from one end only in order to form the shorter electrodes.

A very thin coating of petrolatum shall be used as an adhesive for foil electrodes. A cork or a wax plug provides means for holding mercury in the tube.

Wherever the method of measurement permits, guard rings of foil at both ends of the outer electrode shall be employed.

(d) *Liquids*.<sup>3a</sup>—Some form of cell which will hold the liquid between suitable electrodes is usually employed. Three forms of cells that have been found satisfactory are shown in Appendix III. If the insulation at the bottom of the cell shown in Fig. 19 is perforated the electrode assembly may be used as an immersion unit, which is often convenient. Guard rings (or cylinders) should be used wherever the method of measurement will permit. The electrodes may be either coaxial cylinders or parallel plates. The mechanical construction shall be such that the relative positions of the electrodes

are not altered in the operation of filling the cell with the test liquid, or in bringing the cell and its contents to the desired test temperature. Also, the construction of the cell must be such that in the cleaning operation all traces of a former specimen or cleaning solvent can be completely removed. Any insulation used in the construction of the cell shall be of a material which will not contaminate the test specimen; in the case of a liquid with very low power factor it is desirable to use a cell in which this insulation does not come into contact with the specimen. The electrodes shall not react with the specimen to alter its power factor. (See extended discussion in Appendix III.)

#### Measurements at Other than Room Temperature

14. (a) It is frequently desirable to measure the losses in materials at temperatures other than room temperature. In order to do this the apparatus for holding the specimen may be placed within an oven for elevated temperatures and a refrigerator for lower temperatures, and measurements shall be made while the specimen is in the conditioned atmosphere. In either case the temperature shall be controlled. Proper care shall be exercised in carrying the leads from the bridge to the specimen so that they are properly insulated and shielded. With the susceptance variation method (Section 28) it is usually necessary to place all the apparatus, exclusive of the oscillator and sometimes the vacuum-tube voltmeter, in the temperature chamber. The maximum temperature at which measurements may be made is limited by the type of adhesive used to affix the electrodes, unless silver paint or sprayed metal is used as electrode material.

(b) It is desirable to measure the humidity at all temperatures. Hu-

midity may cause changes as large or larger than those caused by temperature. The percentage relative humidity and the period of time during which the specimen has been exposed to that humidity shall be reported.

## APPARATUS AND PROCEDURE

### Choice of Method

15. The frequency ranges for which the following methods are, at present, deemed suitable, have been tabulated in Section 1. As indicated there the ranges overlap for the several methods and the choice of method in any given situation will usually be determined by the laboratory equipment available, such as resistors, capacitors, detectors, amplifiers, insulating transformers, and the like. All procedures described in these methods are capable of yielding results of the stated accuracy.

### Power Source

16. The generating circuit may be any suitable source which can furnish sufficient current for the specified precision of measurement or voltage gradient in the dielectric. The wave form of the voltage applied to the bridge or existing at the terminals of the resonant circuit shall be approximately of sine-curve shape, having a distortion factor<sup>9</sup> of not more than 5 per cent.

### Detector

17. Any suitable detector may be employed which has the specified sensitivity and which responds only to the frequency of the fundamental component of the voltage applied to the bridge or existing at two terminals of the resonant circuit. These conditions apply also to amplifying means used in conjunction with the detectors and are imposed in conformity with the definition of dielectric phase angle given in Section

2 (b). A selected list of references to detectors in technical literature is given in Appendix IV of these methods.

### Accuracy and Sensitivity

18. These methods contemplate an accuracy of determination of dielectric constant of plus or minus 5 per cent, and an accuracy of determination of power factor of plus or minus 5 per cent but in no case to closer than 0.0001. These accuracies are conditional upon the precision of measurement of the dimensions of the specimen, care in application of electrodes, disposition of the component parts of the test circuits, and calibration of standard resistors and capacitors. The detector used shall have a sensitivity permitting determinations with an apparent accuracy at least twice that specified above.

### General Method of Measurement

19. (a) The diagrams of four of the test circuits (Figs. 7, 9, 10, and 13) in the following sections represent the test capacitor  $C_x$  connected in parallel with a variable precision air capacitor  $C_2$ . The procedure contemplated in these cases is first to secure a reading with the connections as shown, and then to remove from the circuit the specimen capacitor, that is,  $C_x$ ; following this the circuit is restored to its previous condition by changing  $C_2$  and varying the means for changing power factor, the rest of the bridge or resonant circuit remaining unchanged. The effects of various residual errors in other parts of the circuit are thereby minimized. The equivalent parallel capacitance  $C_p$  of the test specimen will then be calculated from the change in reading of the variable precision air capacitor,  $C_2$ .

(b) Frequently, a variable precision air capacitor is not available which can withstand the voltage to be applied to  $C_x$ ; this is usually the case when the high-voltage Schering bridge of Fig. 8 or the transformer bridge of Fig. 12

<sup>9</sup> The distortion factor of a voltage wave is the ratio of the effective value of the residue after the elimination of the fundamental to the effective value of the original wave. Am. Inst. Electrical Engrs., "Electrical Definitions," paragraph 10. 95. 430.

is required. Recourse must then be had to the direct comparison of  $C_x$  with the comparison capacitor  $C_1$ . In such cases, residual errors in the bridge should be minimized or determined by a calibration under conditions as closely similar as possible to those existing when the test specimen was being measured.

### Bridges

20. Five bridge arrangements are shown in Figs. 7, 8, 9, 10, 11, and 12. These bridges have been found satisfactory for the purposes indicated but this is not to be taken as excluding other types of bridges of equal precision and convenience. The formulas for the equivalent parallel capacitance of the specimen  $C_p$ , and for the tangent of its loss angle,  $\tan \delta_p$ , are given in a simplified form which is sufficiently accurate when the loss angles of the various bridge arms are small; the equations are furnished both for the change-of-standard method and for the direct comparison of capacitors without use of the change-of-standard method. When the loss angles of the bridge arms are greater than about 6 deg. ( $\tan \delta = 0.1$ ) or where a precision better than 1 per cent is desired, more elaborate formulas are required. These are given for each bridge in Appendix II.

### Shielding

21. (a) Extensive shielding has been indicated on each bridge as being much the safest procedure when measuring capacitance to  $1\mu\mu\text{f.}$  or better. Shields for all air capacitors are shown connected to the "low-voltage" plate since that is the usual condition under which most variable air capacitors are constructed and calibrated. However, when "three-terminal capacitors" are available, the shield of such capacitors should be connected to the general shielding system. The test capacitor's shield and guard-ring (when one is employed) are connected to the general shielding system

as are also the shields for the resistance units. Leads from the supply or insulating transformer to the bridge, and all bridge leads from terminals *A* and *C* are shielded. Except in the case of the high-voltage Schering bridge (see Fig. 8), the shielding system is grounded. With these precautions it is usually unnecessary to shield the detector circuit. However, when a vacuum-tube amplifier is employed or high precision is sought, it is best to shield the detector circuit also and all low-voltage leads, as indicated in Figs. 8 and 9.

(b) The "Wagner ground" connection is indicated in each bridge (except the high-voltage Schering bridge) as a method to be used to bring detector potential to that of earth. In general, the resistors of the Wagner ground should be of the same order of resistance as the ratio arms,  $R_3$  and  $R_4$ , in order not to load the input transformer more heavily than necessary. It must be possible to adjust the ratio of these resistors with the same precision as the main bridge and for this purpose a low-resistance slide-wire is provided as shown in the figures. The capacitors required for the Wagner ground should have such a range that in their (opposite) extreme positions the difference in capacitance between them will cover any unbalance of stray capacitances between the shields and the measuring circuit. A three-plate type of capacitor is useful for this purpose. A shield between primary and secondary coils of the input transformer should be provided and connected as indicated. A grounded shield, preferably in the form of a metal-screen cabinet or room, enveloping the entire bridge set-up is desirable, especially at frequencies in the broadcast range. This shield and the shield shown about the specimen condenser  $C_x$  should not approach the bridge elements so closely as to increase unduly the capacitance to ground.

(c) The bridge diagrams indicate one side of the detector  $G$  permanently connected at bridge terminal  $D$ , between the ratio arms, with a key provided to connect the other side of  $G$  alternately to the bridge terminal  $B$  and to the shielding system. In proceeding with the test the bridge and Wagner ground are balanced alternately. This procedure is not laborious and is satisfactory for all but the most precise

be made as precisely and completely as the balance of the bridge proper.

### Conjugate Schering Bridge

22. (a) See Fig. 7. This bridge is applicable for all frequencies from 25 cycles per second up to 1.0 megacycle per second.  $R_3$  and  $R_4$  shall be identical resistor units of a type practically free from capacitance, with resistance of the order of 10,000 ohms for use at frequencies of the order of 10 kilocycles per second and less; 1000-ohm units may be used for frequencies of the order of 1 megacycle per second if the capacity of the source warrants. The air capacitor,  $C_3$ , is used to produce a positive reading for  $C_4$  when  $C_2$  alone is in circuit; it should be no larger than is necessary for this purpose.

(b) With the test condenser  $C_x$  in place as indicated in Fig. 7, its switch shall be closed and the bridge balanced by successive adjustments of  $C_2$  and  $C_4$ , alternating with adjustments of the Wagner ground. When  $G$  shows complete balance with the key at  $B$  in either position the readings of  $C_4$  and  $C_2$  shall be recorded. Then the switch of  $C_x$  shall be turned to the shield and new balance values for  $C_4$  and  $C_2$  obtained. From the four values thus secured the equivalent parallel capacitance and the loss angle of the specimen capacitor  $C_x$ , may be calculated from the following approximation formulas; see Appendix II for exact formulas:

$$C_p = C_2 - C'_2 \dots \dots \dots (1)$$

$$D = \tan \delta = \frac{C_2}{C_2 - C'_2} \times \frac{2\pi f R_4 (C'_4 - C_4)}{10^{12}} \quad (2)$$

where:

$C_p$  = equivalent parallel capacitance of the test capacitor formed from the test specimen, in micro-microfarads,

$C'_2$  = capacitance of the variable air capacitor in parallel with  $C_2$ .

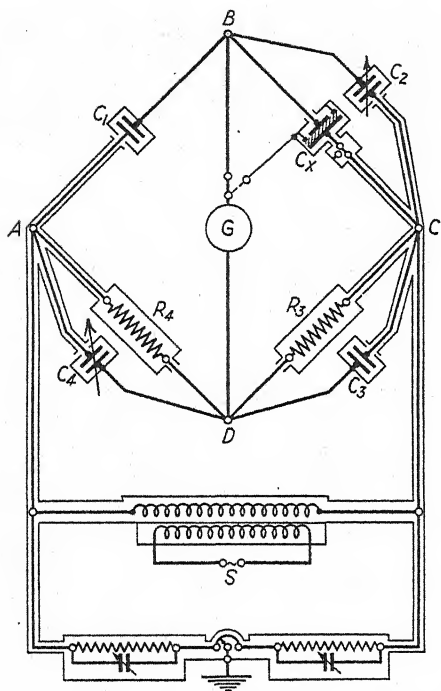


FIG. 7.—Conjugate Schering Bridge (Simple Shielding).

work. For work of the highest precision it becomes essential to keep the detector constantly at ground potential. In that case, one side of  $G$  is permanently attached to the shielding system while the other side is alternately connected to the bridge terminals  $D$  and  $B$  with successive balancings of the Wagner ground and of the bridge, respectively. Under this procedure it is imperative that the balance of the Wagner ground

when the latter is in circuit, in micro-microfarads,

$C_2$  = capacitance of the variable air capacitor when balance has been restored after the test capacitor  $C_x$  has been disconnected from the circuit, in micro-microfarads,

$C'_4$  = capacitance of the capacitor  $C_4$  at balance when  $C_x$  is in circuit, in micro-microfarads,

$C_4$  = capacitance of the capacitor  $C_4$  at balance when  $C_x$  is out of circuit, in micro-microfarads, and

$f$  = frequency, in cycles per second.

The formulas given in Appendix II must be used when both  $C'_4 - C_4$  and  $C_3$  are large.

(c) If the change-of-standard method is not used, dependence may be placed on a single set of observations in a direct comparison of  $C_x$  with  $C_1$ , the corrections to be applied to the observations having been determined by other means. In this case,  $C_2$  shall be removed and  $C_1$  shall be a variable standard air capacitor. At balance, approximately, the following relations hold and the calculations may be made from these formulas; see Appendix II for exact formulas:

$$C_p = C_1 \frac{R_4}{R_3} \dots \dots \dots (3)$$

$$D = \tan \delta = \frac{2\pi f}{10^{12}} \times (R_4 C_4 - R_3 C_3) \dots (4)$$

where:

$R_3$  and  $R_4$  = the resistances of the ratio arms, in ohms,

$C_3$  and  $C_4$  = the corresponding capacitances at balance of the capacitors across  $R_3$  and  $R_4$ , respectively, in micro-microfarads, and

$C_1$  = the capacitance of the variable standard capacitor at balance, in micro-microfarads.

be used when both  $C_4$  and  $C_3$  are large.

### High Voltage Schering Bridge

23. (a) See Fig. 8. This bridge is useful where the voltage required across the specimen capacitor is in excess of about 100 v., and is applicable for all frequencies from 25 cycles per second up to about 10,000 cycles per second, the chief limitations at the higher frequencies being the amplifier used with the detector and the elimination or evaluation of stray capacitance in the resistor  $R_3$ . When  $C_1$  can be made adjustable so that the arms  $R_3$  and  $R_4$  can be identical resistors the method is applicable to frequencies up to 1.0 megacycle per second.  $R_4$  shall be a fixed resistance practically free from capacitance, of such

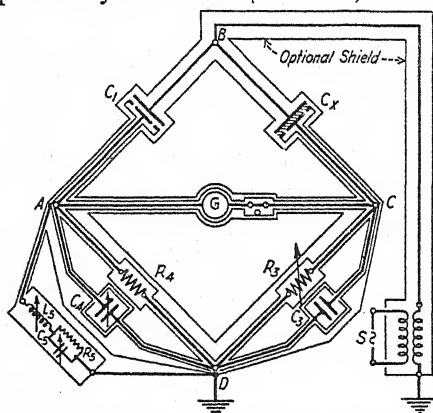


FIG. 8.—High-Voltage Schering Bridge (Complete Shielding).

magnitude as to give a fall of potential across it sufficient to achieve the specified detector sensitivity.  $R_3$  likewise shall be practically free from capacitance; it shall be variable in steps at least as small as 0.1 per cent of its value at balance, not for the precision required but in order that balance may be completed; for measurements of small power factors ability to change  $R_3$  in steps as small as 0.01 per cent of its value will be convenient. The air capacitor  $C_1$  shall be insulated to withstand the voltage to be applied to the test capacitor  $C_x$  without setting up ionization losses.

order of capacitance of  $C_x$ . The air capacitor  $C_3$  may be of the order of 50  $\mu\text{f}$ .

(b) Balance shall be obtained with the switch at  $G$  connected successively to bridge terminal  $C$  and to the shields. Unless the power factor of the stray capacitance to the shields is higher than the power factor of  $C_x$  it will be necessary to use the inductometer  $L_5$  in series with the resistor  $R_5$  rather than the parallel capacitor  $C_5$  in order to balance screen potential. However, it is seldom necessary to balance the inner shields for phase.<sup>10</sup> Also if the capacitance to the shields is small,  $R_5$  may be inconveniently large; in that case a suitable capacitor, with insulation capable of withstanding circuit voltage, may be connected from terminal  $B$  to the inner shield system.

(c) When balance is complete the equivalent parallel capacitance of  $C_x$  and its loss angle may be calculated from Eq. 3 and Eq. 4, respectively, as given in Section 22. It is always wise to demonstrate the accuracy of the bridge readings by substituting, at intervals, for  $C_x$ , a capacitor of known capacitance and loss angle.

### Parallel Resistance Bridge

24. (a) See Fig. 9. This bridge is applicable for all frequencies from 25 cycles per second up to 1.0 megacycle per second.  $R_3$  and  $R_4$  shall be identical resistor units of a type practically free from capacitance, with resistance of the order of 10,000 ohms for use at frequencies of the order of 100 kilocycles per second and less; 1000-ohm units may be used for frequencies of the order of 1.0 megacycle per second if the capacity of the source warrants. The resistor  $R_1$  may be a fixed unit, the value of which is suited to the frequency employed in the measurement and to

the capacitance and conductance of the specimen; it shall be practically free from capacitance.  $R_2$  shall be variable, with a range up to the value of  $R_1$  employed and the change in its capacitance, if appreciable, must be known as its value is altered to restore balance when the test capacitor  $C_x$  has been disconnected.<sup>11</sup>  $R_2$  may be composed with advantage of two parts, one of

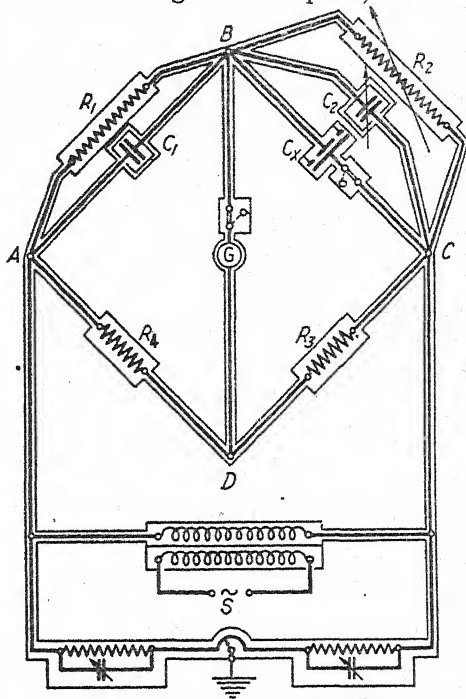


FIG. 9.—Parallel Resistance Bridge (Complete Shielding).

which is variable in steps of 10,000 ohms while the other part is represented in a four-dial or five-dial resistance box.

(b) A balance of both bridge and Wagner ground shall be secured with  $C_x$  in circuit, and then with  $C_x$  out of

<sup>10</sup> W. B. Kouwenhoven and Alfredo Banos, Jr., "High Sensitivity Power Factor Bridge," *Transactions, Am. Inst. Electrical Engrs.*, Vol. 51, p. 202 (1932).

<sup>11</sup> In general, it is desirable to have these resistances at least as large as the reactance of the test condenser; otherwise the bridge loses in sensitivity for the capacitance balance. Also  $R_2$  must be subdivided sufficiently to complete the conductance (power factor) balance. For example: The reactance of 100  $\mu\text{f}$ . at 100 kilocycles per second is 15,916 ohms. If  $R_2$  is of the order of 10,000 ohms, a power factor difference of 0.0001 corresponds to a change in  $R_2$  of about 0.6 ohm and  $R_2$  should have a dial which permits a setting to at least 1 ohm. If  $R_2$  were of the order of only 1000 ohms, a power factor change of 0.0001 would correspond to a change in  $R_2$  of only 0.006 ohm.



circuit. The capacitance and loss angle of  $C_x$  may be calculated from the following approximation formulas; see Appendix II for exact formulas:

$$C_p = C_2 - C'_2 \dots \dots \dots (5)$$

$$D = \tan \delta = \frac{(R'_2 - R_2) \times 10^{12}}{2\pi f R'_2 R_2 (C_2 - C'_2)} \dots (6)$$

where:

$C_p$  = equivalent parallel capacitance of the test capacitor formed from the test specimen, in micro-microfarads,

$C'_2$  = capacitance of the variable air capacitor in parallel with  $C_x$  when the latter is in circuit, in micro-microfarads,

$C_2$  = capacitance of the variable air capacitor when balance has been restored after the test capacitor  $C_x$  has been disconnected from the circuit, in micro-microfarads,

$R'_2$  = resistance at balance of the resistor in parallel with the capacitor  $C_2$ , when  $C_x$  is in circuit, in ohms,

$R_2$  = resistance at balance of the resistor in parallel with the capacitor  $C_2$ , when  $C_x$  is out of circuit, in ohms, and

$f$  = frequency, in cycles per second.

(c) If the change-of-standard method is not used dependence shall be placed on a single set of observations in a direct comparison of  $C_x$  with  $C_1$ , the corrections to be applied to the observations having been determined by other means. In this case,  $C_2$  shall be removed,  $C_1$  shall be a variable standard air capacitor, and the resistance balance shall be secured by varying  $R_1$ . At balance, approximately, the following relations hold and the calculations may be made from these formulas; see Appendix II for exact formulas:

$$C_p = C_1 \frac{R_4}{R_3} \dots \dots \dots (7)$$

$$D = \tan \delta = \frac{10^{12}}{2\pi f} \left( \frac{1}{C_1 R_1} - \frac{1}{C_p R_2} \right) \dots (8)$$

where:

$R_3$  and  $R_4$  = resistances of the ratio arms in ohms,

$R_1$  and  $R_2$  = resistances in parallel with the capacitors  $C_1$  and  $C_x$ , respectively, in ohms, and

$C_1$  = capacitance of the variable standard air capacitor at balance, in micro-microfarads.

### Series Resistance Bridge

25. (a) See Fig. 10. This bridge is applicable for all frequencies from 200 cycles per second up to 1.0 megacycle per

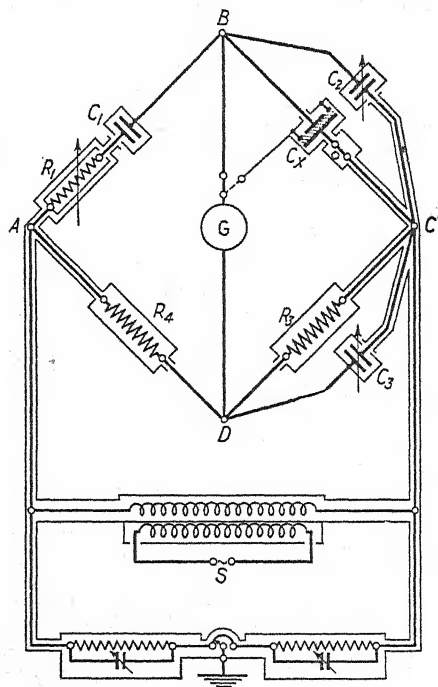


FIG. 10.—Series Resistance Bridge (Simple Shielding).

second.  $R_3$  and  $R_4$  shall be identical resistor units, of a type practically free from capacitance, with resistance of the order of 10,000 ohms for use at frequencies of the order of 100 kilocycles per second and less; 1000-ohm units may be used for frequencies of the order of 1.0

megacycle per second if the capacity of the source warrants. The resistor  $R_1$  shall be practically free from capacitance; it shall be variable up to about 100,000 ohms for frequencies of the order of 200 cycles per second and power factors of the order of 0.02 or smaller; a minimum step of 1 ohm will usually be adequate for this resistance up to frequencies of the order of 100 kilocycles per second. The air capacitor  $C_3$  shall be used to produce a positive reading for  $R_1$  when  $C_2$  alone is in circuit; it shall be no larger than is necessary for this purpose. The variable resistor  $R_1$  is in the "high" side of the bridge; it is shown double-shielded; this will tend to fix its capacitance to earth. Special dial knobs may be required to avoid variable stray capacitance to the hand.

(b) A balance of both bridge and Wagner ground shall be secured with  $C_x$  in circuit and then with  $C_x$  out of circuit. The capacitance and loss angle of  $C_x$  shall be calculated from the following approximation formulas; see Appendix II for exact formulas:

$$C_p = C_2 - C'_2 \dots \dots \dots (9)$$

$$D = \tan \delta = \frac{C_2}{C_2 - C'_2} \times \frac{2\pi f C_1}{10^{12}} (R'_1 - R_1) \dots (10)$$

where:

$C_p$  = equivalent parallel capacitance of the test capacitor formed from the test specimen, in micro-microfarads,

$C'_2$  = capacitance of the variable air capacitor in parallel with  $C_x$  when the latter is in circuit, in micro-microfarads,

$C_2$  = capacitance of the variable air capacitor when balance has been restored after the test capacitor  $C_x$  has been disconnected from the circuit, in micro-microfarads,

$C_1$  = capacitance of the comparison air capacitor, in micro-microfarads,

$R'_1$  = resistance in series with the capacitor  $C_1$  at balance when  $C_x$  is in circuit, in ohms,

$R_1$  = corresponding resistance when  $C_x$  is out of circuit, in ohms, and

$f$  = frequency, in cycles per second.

(c) If the change-of-standard method is not used dependence shall be placed on a single set of observations in a direct comparison of  $C_x$  with  $C_1$ , the corrections to be applied to the observations having been determined by other means. In this case,  $C_2$  shall be removed and  $C_1$  shall be a variable standard air capacitor. At balance, approximately, the following relations hold and the calculations may be made from these formulas; see Appendix II for exact formulas:

$$C_p = C_1 \frac{R_4}{R_3} \dots \dots \dots (11)$$

$$D = \tan \delta = \frac{2\pi f}{10^{12}} (C_1 R_1 - C_3 R_3) \dots (12)$$

where:

$R_3$  and  $R_4$  = resistances of the ratio arms, in ohms, and

$C_3$  = capacitance of the air capacitor across  $R_3$ , in micro-microfarads.

### Transformer Bridge<sup>12</sup>

26. See Figs. 11 and 12. This bridge is applicable for all frequencies from 25 cycles per second (or lower) up to 1000 cycles per second (or higher) for which suitable transformers (phase angles not larger than a few minutes) are available. This method is especially recommended for measurements at high voltage gradients in the specimen and where the highest precision is not required but speed in operation is desirable. Complete shielding is secured in this method without the need for a guard balancing circuit. Only one form of the bridge is

<sup>12</sup> See J. B. McCurley, W. B. Kouwenhoven, and P. H. Dike, "The Transformer Bridge," *Proceedings, Am. Soc. Testing Mats.*, Vol. 35, Part II, p. 734 (1935).



shown; other forms are equally available. Figure 11 is a diagrammatic representation, in the form used for the preceding bridges. Figure 12 shows the shielding arrangements.  $E_3$ ,  $E_4$ , and  $E'_4$  are transformer secondary coils of which the primary coils are excited from the same source; instantaneous polarities of these coils are indicated.

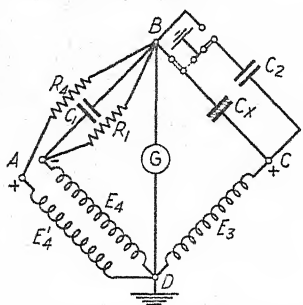


FIG. 11.—Diagrammatic Representation of Transformer Bridge.

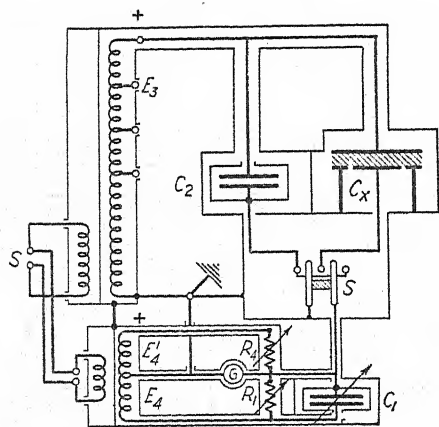


FIG. 12.—Transformer Bridge.

(a) *Three-Balance Method,  $C_2$  Variable.*—When the capacitor  $C_2$  is a variable standard air capacitor, insulated to withstand the voltage to be applied to the test capacitor  $C_x$  without ionization losses setting in, the balancing shall be performed as follows: There are three steps to the balancing operation. With  $C_x$  in circuit and  $C_2$  grounded,  $R_1$  shall be set at 10,000 ohms (100,000 ohms) or any convenient value; balance shall

be secured by varying  $C_1$  and  $R_4$ . This may be called the preliminary balance. Next  $C_x$  shall be grounded while  $C_2$  is put into circuit. Balance shall be secured by varying  $C_2$  and  $R_4$ ;  $C_1$  shall be left at the "preliminary" setting. At the third balance, the switch  $S$  shall be returned to its first position with  $C_x$  in circuit and  $C_2$  grounded; balance shall be secured only by a change in  $R_1$ . Then, with only a very small degree of approximation (see Appendix II):

$$C_p = C_2 \dots \dots \dots (13)$$

$$D = \tan \delta = \frac{(R'_1 - R''_1) \times 10^{12}}{2\pi f C'_1 R'_1 R''_1} \dots \dots (14)$$

where:

- $C_p$  = equivalent parallel capacitance of the test capacitor formed from the test specimen, in micro-microfarads,
- $C_2$  = capacitance of the variable standard air capacitor, in the second balance, in micro-microfarads,
- $C'_1$  = capacitance of the "low-voltage" capacitor, unchanged after the first balance, in micro-microfarads,
- $R'_1$  = resistance of the "negative" resistor, as selected for the first balance, in ohms,
- $R''_1$  = resistance of the "negative" resistor in the third balance, in ohms, and
- $f$  = frequency, in cycles per second.

(b) *Two-Balance Method,  $C_2$  Fixed.*—When there is not available a variable standard air capacitor capable of withstanding the test voltage, a fixed value standard air capacitor shall be employed for  $C_2$ . Two balances only will be required in this case. The first shall be made with  $C_x$  in circuit;  $R_1$  shall be set at 10,000 ohms (100,000 ohms) or any convenient value; balance shall be secured by varying  $C_1$  and  $R_4$ . Next  $C_2$

shall be put into circuit,  $C_s$  being grounded and balance shall be secured by varying  $C_1$  and  $R_1$ . Then, with only a small degree of approximation (see Appendix II):

$$C_p = C_2 \frac{C'_1}{C_1} \dots \dots \dots (15)$$

$$D = \tan \delta = \frac{10^{12}}{2\pi f} \left( \frac{1}{C'_1 R'_1} - \frac{1}{C''_1 R''_1} - \frac{K}{R'_4 C'_1} + \frac{K}{R'_4 C''_1} \right) \dots \dots \dots (16)$$

where:

$C_p$  = equivalent parallel capacitance of the test capacitor formed from the test specimen, in micro-microfarads,

$C_2$  = capacitance of the standard air capacitor, in micro-microfarads,

$C'_1$  = capacitance of the "low-voltage" capacitor in the first balance, in micro-microfarads,

$C''_1$  = capacitance of the "low-voltage" capacitor in the second balance, in micro-microfarads,

$R'_1$  = resistance of the "negative" resistor in the first balance, in ohms,

$R''_1$  = resistance of the "negative" resistor in the second balance, in ohms,

$R'_4$  = resistance of the "positive" resistor in the first balance, in ohms,

$f$  = frequency, in cycles per second, and

$K = E'_4/E_4$ , the ratio of the "positive" voltage to the "negative" voltage. This ratio is usually near unity, although it may have other values especially when it is desirable to secure a more convenient value for  $R_4$ . When  $K$  is near unity its value may be secured from

two successive comparisons of  $C_2$  and  $C_1$ , before and after reversing the primary and secondary connections so that  $C_1$  is supplied first from the "negative" voltage side of the transformer (as shown in Fig. 12) and then from the "positive" side (see Appendix II).

### Resonant Circuit, Resistance Variation Method

27. (a) See Fig. 13. This method is applicable for frequencies from about 100 kilocycles per second up to 1.0 megacycle

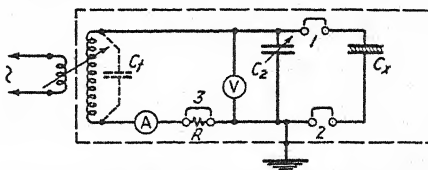


FIG. 13.—Resonant Circuit, Resistance Variation Method.

per second. The oscillator shall have a power rating sufficient to prevent any large reaction on frequency or voltage output when coupled to the measuring circuit. Several coupling coils will be needed when test measurements are to be made over a range of frequencies. The capacitor  $C_2$  shall have a range up to say 200  $\mu\mu\text{f.}$  and shall be adjustable to 0.5  $\mu\mu\text{f.}$  or better. It may be necessary, at some frequencies, to supplement  $C_2$  with a second capacitor  $C_1$ , (shown in dotted line in Fig. 13) connected across the terminals of the coupling coil. The switches 1, 2, and 3 shall be formed with short links of copper wire dipping into mercury cups mounted on rods or tubes, 4 to 6 in. (10 to 15 cm.) in length, of quartz or high-resistivity glass. The resistors represented by  $R$  in Fig. 13 shall be short lengths of high-resistivity nonmagnetic wire (advance or manganin is suitable) sealed into glass tubes for convenience in handling, arranged to

bridge the mercury cups of switch 3. For most work a range of resistance from 0.5 ohm to 20 or 30 ohms will be adequate.

(b) The indicator of resonance and of the current strength may be either an ammeter  $A$ , or a vacuum tube voltmeter  $V$ . Both are shown in Fig. 13. The ammeter  $A$  shall consist of a low-resistance heater, preferably of less than 2 ohms resistance, with a thermocouple which used in conjunction with a short-period low-resistance reflecting galvanometer shall give a full scale deflection for a current in the order of 10 to 20 ma. The vacuum tube voltmeter shall have reasonably small interelectrode capacitance and losses. The specimen shall be mounted on insulating pillars to reduce stray capacitance of adjacent objects and the "high" lead from switch 1 to the "high" electrode shall be as short as possible. The measurements shall be carried out inside a metal screen shielding enclosure and it is usually desirable to interpose a grounded screen between the coupling coil and the oscillator which serves as the source.<sup>13</sup>

(c) For the first step, a suitable coupling coil shall be selected, switches 1, 2, and 3 shall be closed with the copper links, the source oscillator shall be brought to the desired frequency and the resonant circuit tuned by varying  $C_2$  and also by introducing  $C_x$  if necessary. The coupling between the measuring circuit and the source shall then be altered to give a satisfactorily large deflection of the ammeter or voltmeter, whichever is being used. A careful

adjustment of  $C_2$  for perfect resonance shall then be made and the reading of the ammeter or voltmeter recorded. The links at switches 1 and 2 shall now be removed, the link at switch 3 shall be replaced with a resistor, say of 10 ohms. Resonance shall be again secured by varying  $C_2$ . A value of  $R$  shall then be inserted which will give a slightly larger deflection than when  $C_x$  was in circuit; this shall be followed by other values of  $R$  to give deflections as nearly equal as possible to that obtained with  $C_x$ , and other values of  $R$  to give somewhat smaller deflections. A slight readjustment of  $C_2$  may be necessary and shall be recorded. The values of  $R$  and  $C_2$  which will give the same deflection as with  $C_x$  in circuit shall then be obtained by interpolation.

$$C_x = \frac{C_2}{1 + D^2} - C'_2 \dots \dots \dots (17)$$

$$D = \tan \delta = \frac{C_2}{C_2 - C'_2} \times 2\pi f C_2 R_i \times 10^{-12} \dots (18)$$

where:

$C_p$  = equivalent parallel capacitance of the test capacitor formed from the test specimen, in micro-microfarads,

$C'_2$  = first resonant value of standard capacitor with  $C_x$  in circuit, in micro-microfarads,

$C_2$  = second resonant value with  $C_x$  out of circuit, in micro-microfarads,

$R_i$  = interpolated value of series resistance to give same resonant current (or voltage) as with  $C_x$  in circuit, in ohms, and

$f$  = resonant frequency, in cycles per second.

#### Resonant Circuit, Susceptance Variation Method

28. (a) This method has been found satisfactory for frequencies from as low

<sup>13</sup> The series inductance and resistance of the leads in the circuit of  $C_x$  from the junction with the circuit of  $C_2$  may produce serious errors. The leads should be kept as short as possible and form as small a loop as possible. If several values of capacitance, which have been otherwise standardized, can be inserted in the place of the specimen capacitor,  $C_x$ , with no other change in the configuration of the circuit, the inductance and resistance of the leads may be determined. The inductance and equivalent resistance of the circuit of the variable standard capacitor,  $C_2$  will sometimes require evaluation at the higher frequencies. See paper by R. F. Field and D. B. Sinclair, "A Method for Determining the Residual Inductance and Resistance of a Variable Air Condenser at Radio Frequencies," *Proceedings, Inst. Radio Engrs.*, Vol. 24, p. 255 (1936).

as 10 kilocycles per second up to 100 megacycles per second, and is especially recommended for use at frequencies above 1.0 megacycle per second. The method makes use of the fact that the width of the resonance curve of a tuned circuit depends on the circuit conductance so that it is possible to determine the equivalent parallel circuit conductance (and power factor) from measurements, first of the resonance capacitance, and then of the capacitance changes to both sides of resonance required to reduce the voltage across the capacitor to a chosen fraction of the resonance voltage. The loss angle of the circuit is then defined by the equation:

$$D = \tan \delta = \frac{\Delta C P}{2C\sqrt{1 - P^2}} \dots (19)$$

where:

$C$  = capacitance in the circuit at resonance,

$\Delta C$  = total change in capacitance required to detune to a voltage  $V$  first on one side and then on the other side of the resonance voltage  $V_r$ , and

$$P = V/V_r$$

When the voltage ratio is chosen as 0.707,  $\tan \delta = \frac{\Delta C}{2C}$ .

These readings will yield the loss angle of the total circuit being measured in which the test specimen capacitor has been included. A second set of observations shall now be made with the test specimen capacitor removed and with the circuit retuned with no other change in the circuit than the adjustments of a variable capacitor to reestablish resonance and to give the two off-resonance capacitances at the fractional voltage. The equivalent conductance (and power factor) of this remaining measuring circuit shall then be calculated in accordance with Eq. 19. The capacitance

and the parallel conductance and power factor of the specimen capacitor shall be obtained by difference; see Paragraphs (h) and (i).

The inductances of the circuits containing the specimen capacitor and the variable standard capacitor become increasingly troublesome as the measuring frequency is increased.<sup>13</sup> If the variable standard capacitor used is a two-plate capacitor, with plate diameters equal to the diameter of the specimen capacitor, then the first set of readings may be taken with the specimen capacitor held between the plates of the variable standard capacitor and the second set of

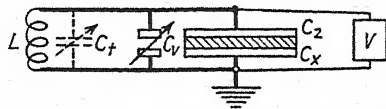


FIG. 14.—Resonant Circuit, Susceptance Variation Method.

readings, with the specimen removed, may be taken with the plates of the variable standard brought together to reestablish resonance. By this procedure the geometry of the resonant circuit is practically identical in the two sets of measurements and the troublesome effects of inductance, resistance, and change in stray capacitance may be kept smaller than the desired limit of accuracy up to frequencies above 100 megacycles per second. Although it is understood that this procedure may not be essential at frequencies of the order of 1 megacycle per second and lower, the following Paragraphs (b) to (j) describe in detail the procedure for use at the higher frequencies.

(b) The measuring circuit (see Fig. 14) shall consist of an inductor  $L$  and a calibrated variable parallel-plate capacitor  $C_2$ , in which the plates have a diameter of 2.0 in. This capacitor also serves as a holder for the specimen and shall be sufficiently rigid in construction to resist distortion when holding the specimen.

An additional variable air capacitor  $C_1$  shall be used to tune the circuit to the specified frequency, otherwise  $L$  shall be selected of the order of inductance required for the frequency level desired and the oscillator source shall be adjusted to resonate the test circuit. One and sometimes two vernier air capacitors  $C_v$  are useful for detuning the circuit to the chosen voltage ratio  $V/V_r$ . The fixed ("high") plates of  $C_2$  and  $C_v$  should be common, if possible, while their movable plates shall be effectively grounded. Also the movable plates of these two capacitors shall be mounted on screws with a fine thread. The conventional micrometer head having 40 threads to the inch has been found mechanically satisfactory, but to promote stable electrical performance it may be necessary to shunt the thread contact with a low and constant impedance path such as a mercury well or a flexible copper screen. A parallel-plate capacitor with electrode diameters of 2 in. will have a useful capacitance range from about 70.7  $\mu\text{mf.}$  at 10 mils to 7.07  $\mu\text{mf.}$  at 100 mils separation. To obtain high sensitivity the " $Q$ " ( $Q = \omega L/R$ ) of the inductance coil used shall be at least 200. The residual circuit capacitance shall be kept at a minimum and the insulating supports used for parts not at earth potential shall have low dielectric loss.

(c) Although the capacitance of the test capacitor  $C_t$  is practically equal to the "geometric" capacitance of the parallel-plate capacitor  $C_2$  as calculated from the area of its plates and their spacing at the second resonance setting according to the formula in Section 7 (b), there is some change with spacing in the edge effect of the plates which cannot be ignored when thick plates are used, and there is the likelihood that lack of parallelism of the plates even with the most careful machine work will become troublesome at the very small spacings

frequently required for resonance after removal of the specimen. It is well therefore to carry out a calibration of  $C_2$  by comparison at an audio frequency with a suitable precision capacitor, taking frequent observations during the last revolution of the movable plate before contact. In such a calibration, the capacitance assigned to a setting corresponding to the thickness of the specimen should be taken as the capacitance calculated from the dimensions, any deviation from this value being considered as a fixed, stray capacitance which is part of  $C_t$ . As a practical procedure, however, and in order to secure a fixed calibration curve, the capacitance assigned to a setting of 100 mils shall be that calculated from the dimensions, and the remainder of the calibration shall be adjusted accordingly.

(d) A vacuum-tube voltmeter having a low input capacitance and power factor shall be used as the resonance indicator. Approximate values for a satisfactory voltmeter are a capacitance of 10  $\mu\text{mf.}$  and a power factor of 0.01. Although it is not necessary that the instrument shall register correctly the voltage in volts, at its terminals, it is essential that the relative voltage scale of the instrument shall be known with the desired precision. A vacuum tube with a low input capacitance, such as the "acorn" type of tube, may be employed in either diode or triode connection.

(e) The oscillator may be any suitable vacuum-tube generator of sufficient stability and power output so that the frequency, in particular, and to a less extent the amplitude at the output terminals, shall not vary with temperature, time, small fluctuations in supply voltage, or with the change in loading conditions which occurs during the measuring period of each specimen. Resistive, capacitive, or inductive coupling may be employed. The degree of coupling shall be such that the reaction of the tuned

circuit on the oscillator is negligible. In any case increasing the degree of coupling has the effect of increasing the effective resistance of the resonant circuit. In general  $\tan \delta$  with specimen removed shall not exceed 0.01 and should be made as small as possible.

(f) The oscillator, measuring circuit, and vacuum-tube voltmeter shall be individually screened and shall be mounted on a copper plate to which the measuring circuit is connected by a short massive copper conductor. Connections between the three units shall be completely screened using, for example, concentric-conductor leads.

(g) The measurement may be made by either of the two procedures described in Paragraphs (h) and (i).

(h) *Change-of-Capacitance Procedure.*—The specimen capacitor, with electrodes affixed, shall be clamped in the parallel-plate capacitor holder,  $C_2$  and the circuit resonated at the desired frequency by varying  $C_i$  (or it may be sufficient to tune the oscillator to the circuit). The maximum voltage  $V_1$  shall be noted and the vernier condenser  $C_v$  shall be then slowly changed until the voltage is reduced to  $0.707 V_1$ , first on one side, then on the other, of the resonant point. The corresponding capacitance values  $C_1$  and  $C'_1$  shall be recorded.  $C_v$  shall be restored to resonance setting.

After removing the specimen, the circuit again shall be returned to resonance by means of the parallel-plate capacitor  $C_2$ , the oscillator and  $C_i$  remaining undisturbed. The maximum voltage  $V_o$  shall be noted and the capacitance value,  $C_r$  of the parallel-plate capacitor shall be recorded. The capacitance change to reduce the circuit voltage to  $0.707 V_o$  shall be determined by detuning on each side of resonance as before and the corresponding vernier capacitances  $C_o$  and  $C'_o$  shall be recorded.

Then:

$$\Delta C_1 = C_1 - C'_1 \quad \Delta C_o = C_o - C'_o$$

$$D = \tan \delta = \frac{\Delta C_1 - \Delta C_o}{2C_p} \dots \dots \dots (20)$$

$$C_p = C_r - C'_2 + C'_a \dots \dots \dots (21)$$

where:

$C_p$  = capacitance of the specimen, in micro-microfarads,

$C_r$  = calibration capacitance of the parallel-plate capacitor  $C_2$  at the second resonance adjustment,

$C'_2$  = calibration capacitance of the parallel-plate capacitor  $C_2$  at a setting equal to the measured thickness of the specimen dielectric, and

$C'_a$  = calculated capacitance of  $C_2$  at a setting equal to the measured thickness of the specimen. If  $S$  (in inches) is the thickness of the specimen and  $A$  (in square inches) is the plate area,

$$C'_a = 0.2249 \frac{A}{S}.$$

Equation 21 is intended to take care of the change in edge correction capacitance, inequality in screw thread, and eccentricity of plates, which occur as the plates of  $C_2$  are advanced for the second resonance adjustment. If the thickness of the specimen  $S$  happens to be 0.100 in.,  $C'_2 = C'_a$ , and for this particular case,  $C_p = C_r$  (see Paragraph (c)).

(i) *Change-of-Voltage Procedure.*—This procedure makes use of the resonant voltages of the circuit with the specimen "in" and "out," and of the  $\Delta C$  required to detune the circuit to 0.707 voltage on either side of the resonant voltage with the specimen "out." The procedure is particularly satisfactory when the capacitance setting of the parallel-plate capacitor  $C_2$  can be read to a satisfactory degree of precision for the determination of  $\Delta C$ , because the vernier capacitor  $C_v$  is not then required. A close spacing of the parallel plate electrodes or a very low power factor of the circuit with the specimen removed will require the use of the vernier capacitor  $C_v$ .



The specimen with its electrodes shall be clamped between the plates of the capacitor  $C_2$  and the circuit tuned to resonance at the desired frequency by means of  $C_1$ , or the oscillator source shall be tuned to the resonant circuit. The resonant voltage  $V_1$  shall be recorded. Next the specimen shall be removed and the circuit retuned to resonance by means of  $C_2$ . The maximum resonant voltage  $V_o$  and the capacitance value  $C_r$  of the parallel-plate capacitor shall be recorded. Then the circuit shall be detuned on each side of resonance to reduce the voltage to  $0.707 V_o$ , and the corresponding capacitance values  $C_o$  and  $C_o'$  recorded. From these observations:

$$D = \tan \delta = \frac{\Delta C_o}{2C_p} \times \frac{V_o - V_1}{V_1} \dots (22)$$

where:

$$\begin{aligned} \Delta C_o &= C_o - C_o', \text{ and} \\ C_p &= C_r - C_2' + C_a' \\ &\quad \text{as in Eq. 20.} \end{aligned}$$

(j) The desirability of using a specimen which has the same diameter as the plates of the capacitor holder  $C_2$  is indicated in Paragraph (a). The foil electrodes of the specimen shall come to the edge. Satisfactory measurements can also be made when the specimen is smaller than the plates of the capacitor  $C_2$ . This becomes necessary when the specimen is very thin or has a large value of dielectric constant. The procedure in that case and the formula for  $\tan \delta$  are the same as those described in Paragraphs (h) and (i). The expression for the specimen capacitance is now:

$$C_p = C_r - C_2'' + C_a'' \dots (23)$$

where:

$C_2''$  = calibration capacitance of the parallel-plate capacitor  $C_2$  at a setting equal to the measured thickness of the specimen, and  
 $C_a''$  = calculated "air-capacitance" of the specimen.

$$C_a'' = 0.2249 \frac{A_p}{S}$$

$A_p$  = area of the specimen in square inches, and

$S$  = measured thickness of the specimen dielectric in inches.

Use of a specimen larger than the plates of  $C_2$  is not recommended since the edge correction with the specimen between the plates would be different from that which exists when the specimen has been removed.

### Resonant Circuit, Resonance Rise Method

29. (a) This method has been found suitable for frequencies from 50 kilocycles per second up to 10 megacycles per second. The method makes use of the fact that when a known radio-frequency voltage is introduced into a resonant circuit the ratio of this voltage to the voltage appearing across either the inductor or the capacitor of the resonant circuit is approximately the dissipation factor of the circuit. While this voltage can be introduced into the circuit by inductive, capacitive, or resistive coupling, its magnitude is more easily measured for resistive coupling, as shown in Fig. 15.

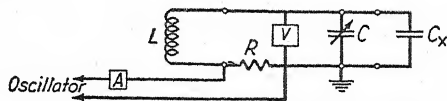


FIG. 15.—Resonant Circuit, Resonance Rise Method.

A radio-frequency oscillator furnishes to resistor  $R$  a constant current which is measured by radio-frequency ammeter  $A$ . This resistor shall be designed to have a very small reactance so that its impedance will differ from its resistance, at the highest operating frequency, by a negligible amount.<sup>14</sup>

<sup>14</sup> A commonly used value of resistance is 0.04 ohm for which the inductance can be reduced to a value of 0.07 mμh. The ratio of impedance to resistance at 10 megacycles is then 1.006.

The resonant circuit is composed of fixed inductor  $L$  and variable air capacitor  $C$ . From the vacuum tube voltmeter  $V$  is read the voltage developed across capacitor  $C$ . The dissipation factor of the entire circuit is the ratio of the voltage drop across resistor  $R$  due to current  $I$  from the oscillator, to the voltage  $E$  developed across capacitor  $C$ .

$$D_1 = \tan \delta = \frac{IR}{E} \dots \dots \dots (24)$$

(b) When all components required for this measurement (oscillator, ammeter  $A$ , resistor  $R$ , and vacuum-tube voltmeter  $V$ ) are assembled in a single unit, the vacuum-tube voltmeter,  $V$ , can be calibrated directly in terms of dissipation factor  $D^{15}$  by choosing a standard oscillator current  $I$  and always adjusting the current as indicated by ammeter  $A$  to this value.

(c) The vacuum-tube voltmeter, oscillator, and tuning capacitor shall in general conform to the requirements prescribed in Section 28 (d), (e), and (f).

(d) The specimen capacitor, with electrodes affixed, shall be connected by short low-resistance leads to the terminals of capacitor  $C$ . A suitable inductor  $L$  shall be selected which will permit resonance at the chosen frequency. The capacitance of capacitor  $C$  and the reading of vacuum-tube voltmeter  $V$  at resonance shall be designated  $C_2$  and  $D_2$ , respectively.

The contact to the ungrounded electrode of the test specimen shall be broken by raising the connecting wire a small amount (about  $\frac{1}{4}$  in. if the wire is bare and of small diameter). Resonance shall be reestablished by adjusting capacitor  $C$ , and the new readings of capacitor and voltmeter,  $C_1$  and  $D_1$ , shall be noted.

Then:

$$D = \tan \delta = \frac{(D_2 - D_1)C_1}{C_1 - C_2} \dots \dots (25)$$

$$C_p = C_1 - C_2 \dots \dots \dots (26)$$

where:

$C_p$  = capacitance of the specimen, in micro-microfarads,

$C_1$  = capacitance of the standard air capacitor with the specimen out of circuit, in micro-microfarads,

$C_2$  = capacitance of the standard air capacitor with the specimen in circuit, in micro-microfarads,

$D_1$  = dissipation factor indicated by voltmeter with specimen disconnected, and

$D_2$  = dissipation factor indicated by voltmeter with specimen connected in parallel with standard capacitor.

NOTE.—When the voltmeter is calibrated in terms of storage factor  $Q$ , Eq. 25 becomes:

$$D = \frac{1}{Q} = \frac{Q_1 - Q_2}{Q_1 Q_2} \times \frac{C_1}{C_1 - C_2}$$

where:

$Q_1$  = storage factor indicated by voltmeter with sample disconnected, and

$Q_2$  = storage factor indicated by voltmeter with sample connected in parallel with standard capacitor.

Since this expression for dissipation factor of the specimen depends on the difference between two observations of dissipation factor, ( $D_2 - D_1$ ), the inductor  $L$  should be so chosen as to have as small a dissipation factor as possible. It should be possible to obtain inductors with dissipation factors not larger than 0.003 (storage factors not smaller than 330) in the frequency range for which this method is recommended.

## REPORT

### Report

30. The report shall include the following:

<sup>15</sup> The voltmeter is frequently calibrated in terms of storage factor  $Q$ , which is the reciprocal of  $D$ , which provides an approximately uniform scale.



(a) A description of the material; that is, the name, grade, and color; and the name of the manufacturer,

(b) The following test conditions: the frequency in cycles (or kilocycles or megacycles) per second; temperature of the atmosphere in degrees Centigrade; percentage relative humidity of the atmosphere; the conditioning of the specimen (hours at humidity and temperature); the

kind of electrodes; and the voltage gradient in the dielectric while under test,

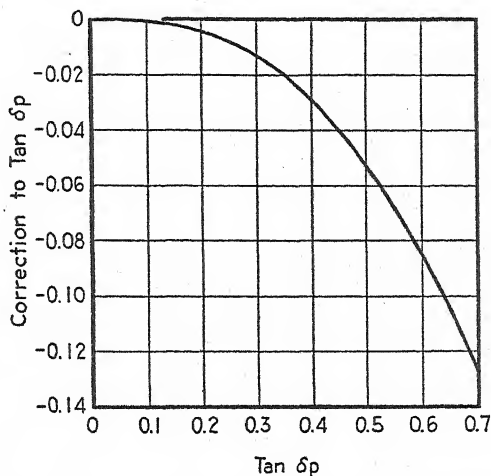
(c) The following values for the specimen: the capacitance of the specimen in micro-microfarads; effective area of the specimen electrodes; the average thickness of the specimen between electrodes,

(d) The power factor, the dielectric constant, and the loss factor of each specimen, and

(e) Method of measurement.

(See Appendices, pp. 673 to 681)

## APPENDIX I

CORRECTIONS TO BE APPLIED TO  $\tan \delta_p$  TO SECURE  $\cos \theta_p$ FIG. 16.—Corrections to be Applied to  $\tan \delta_p$  to Secure  $\cos \theta_p$ .

## APPENDIX II

## EXACT FORMULAS

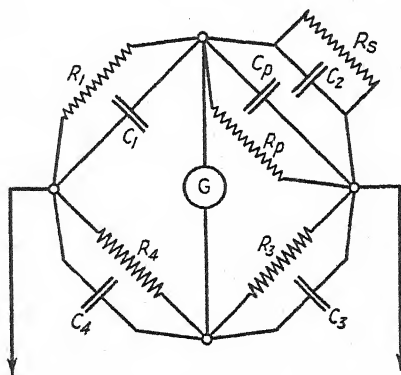


FIG. 17.—Generalized Diagram Showing Capacitors With Conductance and Resistors With Capacitance.

## I. Conjugate Schering Bridge:

$C_2R_5$  represents the standard air capacitor.

$C_1R_1$  represents the comparison air capacitor.

$C_pR_p$  represents the test capacitor.

The capacitors  $C_3$  and  $C_4$  across the ratio arms  $R_3$  and  $R_4$  contain the stray capacitances of those resistors as well as the capacitances of the actual devices.

Two successive balances of the bridge shall be made, with and without the test capacitor  $C_x$  in place. From these the following are obtained:

$$C_p = \frac{C_2 (1 + \tan \gamma_4 \tan \delta_2)}{(1 + \tan \gamma'_4 \tan [\delta_2 + \gamma'_4 - \gamma_4])} - C'_2 \dots \dots \dots (1)$$

$$\tan \delta_p = \left(1 + \frac{C'_2}{C_p}\right) \tan [\delta_2 + \gamma'_4 - \gamma_4] - \frac{C'_2}{C_p} \tan \delta'_2 \dots \dots \dots (2)$$

where  $\tan \delta_2 = \frac{1}{\omega C_2 R_2}$  = power factor (approximate) of  $C_2$  at second setting,

$\tan \delta'_2 = \frac{1}{\omega C'_2 R'_2}$  = power factor (approximate) of  $C_2$  at first setting,

$\tan \gamma_4 = \omega C_4 R_4$  and

$\tan \gamma'_4 = \omega C'_4 R_4$ .

## II. High-Voltage Schering Bridge:

Standard capacitor  $C_2$  not used; same as Conjugate Schering Bridge with direct comparison.

$$C_p = \frac{R_1}{R_2} C_1 \frac{(1 + \tan \gamma_3 \tan \delta_1)}{(1 + \tan \gamma_4 \tan [\gamma_4 + \delta_1 - \gamma_3])} \dots \dots \dots (3)$$

$$\tan \delta_p = \tan (\gamma_4 + \delta_1 - \gamma_3) \dots \dots \dots (4)$$

where  $\tan \delta_1 = \frac{1}{\omega C_1 R_1}$  = power factor (approximate) of  $C_1$ ,

$\tan \gamma_3 = \omega R_3 C_3$ , and

$\tan \gamma_4 = \omega R_4 C_4$ .

If the capacitances of  $R_3$  and  $R_4$  are negligible,  $C_3$  and  $C_4$  are the readings of the capacitors connected across them.

## III. Parallel Resistance Bridge:

Diagram same as for Conjugate Schering Bridge except additional variable parallel resistance  $R_2$  connected across  $C_p R_p$ . Two successive balances of the bridge shall be made, with and without the test capacitor  $C_x$  in place. From these the following are obtained:

$$C_p = C_2 - C'_2 \dots \dots \dots (5)$$

$$\tan \delta_p = \tan \delta_m - \tan \delta'_m + \frac{C_2}{C_p} \tan \delta_2 - \frac{C'_2}{C_p} \tan \delta'_2 \dots \dots \dots (6)$$

where  $\tan \delta_2 = \frac{1}{\omega C_2 R_2}$  = power factor (approximate) of  $C_2$  at second balance position,

$\tan \delta'_2 = \frac{1}{\omega C'_2 R'_2}$  = power factor (approximate) of  $C_2$  at first balance position,

$\tan \delta_m = \frac{1}{\omega C_p R_2}$ , and

$\tan \delta'_m = \frac{1}{\omega C_p R'_2}$ .

If the change-of-standard method is not employed but dependence is placed on a single comparison with the standard air capacitor  $C_1$ , then:

$$C_p = \frac{R_4}{R_3} C_1 \frac{(1 + \tan \gamma_3 \tan \delta_1)}{(1 + \tan \gamma_4 \tan [\delta_1 + \gamma_4 - \gamma_3])} \dots \dots \dots (7)$$

$$\tan \delta_p = \tan (\delta_1 + \gamma_4 - \gamma_3) - \tan \delta_m \dots \dots \dots (8)$$

where the loss angles have the same significance as above.

#### IV. Series Resistance Bridge:

Diagram same as for Conjugate Schering Bridge, except  $C_1$  and  $R_1$  shall be connected in series instead of in parallel. Two successive balances of the bridge shall be made with and without the test capacitor  $C_x$  in place. From these the following are obtained:

$$C_p = C_2 \frac{(1 + \tan \beta_1 \tan \delta_2 + [\tan (\delta_2 + \gamma_3 - \beta_1)] [\tan \delta_2 - \tan \beta_1])}{(1 + \tan \beta'_1 \tan (\delta_2 + \beta'_1 - \beta_1) + [\tan (\delta_2 + \gamma_3 - \beta_1)] [\tan (\delta_2 + \beta'_1 - \beta_1) - \tan \beta'_1]) - C'_2} \dots \dots \dots (9)$$

$$\tan \delta_p = \left(1 + \frac{C'_2}{C_p}\right) \tan (\delta_2 + \beta'_1 - \beta_1) - \frac{C'_2}{C_p} \tan \delta'_2 \dots \dots \dots (10)$$

The various loss angles in Eq. (9) and Eq. (10) are defined by the following:

$$\tan \beta_1 = \omega C_1 R_1 \qquad \tan \gamma_3 = \omega C_3 R_3$$

$$\tan \beta'_1 = \omega C_1 R'_1$$

$$\tan \delta_2 = \frac{1}{\omega C_2 R_2} = \text{power factor (approximate) of } C_2 \text{ at second balance position, and}$$

$$\tan \delta'_2 = \frac{1}{\omega C'_2 R'_2} = \text{power factor (approximate) of } C_2 \text{ at first balance position.}$$

If the change-of-standard method is not employed but dependence is placed on a single comparison with the standard air capacitor  $C_1 R_1$ , then:

$$C_p = \frac{R_4}{R_3} C_1 \frac{1}{(1 + \tan \beta_1 \tan (\beta_1 + \gamma_4 - \gamma_3) + \tan \gamma_4 [\tan (\beta_1 + \gamma_4 - \gamma_3) - \tan \beta_1])} \dots \dots \dots (11)$$

$$\tan \delta_p = \tan (\beta_1 + \gamma_4 - \gamma_3) \dots \dots \dots (12)$$

where the loss angles have the same significance as above, with

$$\tan \gamma_4 = \omega C_4 R_4$$

#### V. Transformer Bridge:

(a) *Three-Balance Method,  $C_2$  Variable.*—The exact formulas containing a term which takes into account the phase difference between the "positive" voltage  $E'_4$ , and the "negative" voltage  $E_4$ , are quite cumbersome.

$$C_p = C_2 (1 + A) \dots \dots \dots (13)$$

$$\tan \delta_p = \frac{1}{(1 + A)} \left( \tan \delta_2 + \frac{(\tan \delta'''_1 - \tan \delta'_1) (1 - P + \tan \delta_2 \tan \delta_2)}{(1 - P)^2 + \tan^2 \delta_2} \right) \dots \dots \dots (14)$$

where  $\tan \delta_2 = \frac{1}{\omega C_2 R_2}$  = power factor (approximate) of the capacitor  $C_2$ ,

$$\tan \delta'''_1 = \frac{1}{\omega C_1 R'''_1} \qquad \tan \delta'_1 = \frac{1}{\omega C_1 R'_1}$$

$$\tan \delta_4 = \frac{1}{\omega C_1} \left( \frac{1}{R_3} + \frac{1}{R'_1} - \frac{K}{R''_4} \right) \qquad K = \frac{E'_4}{E_4}$$

$$\frac{1}{\omega C_1 R_s} = \text{power factor (approximate) of the capacitor, } C_1$$

$$P = \frac{K \tan \delta_s}{\omega C_1 R''_4} \quad \text{where } \delta_s = \text{phase difference between } E'_4 \text{ and } E_4$$

$$A = \frac{(\tan \delta''_1 - \tan \delta'_1) (\tan \delta_s - [1 - P] \tan \delta_s)}{(1 - P)^2 + \tan^2 \delta_s}$$

It is obvious that the values of the resistors  $R_1$  and  $R_4$  must be kept high if the correction terms in these quantities are to have negligible values.

(b) *Two-Balance Method,  $C_2$  Fixed.*—The complete expressions are quite lengthy and contain terms in  $\left(1 - \frac{K \tan \delta_s}{C'_1 R'_4}\right)$  and  $\left(1 - \frac{K \tan \delta_s}{C''_1 R'_4}\right)$

where  $\delta_s$  is the phase difference between the voltages  $E_4$  and  $E'_4$ . If  $\delta_s$  is so small that these expressions differ from unity by negligible amounts, then:

$$C_p = C_2 \frac{C'_1}{C''_1} \frac{(1 - \tan \delta_s \tan [\delta'_s - \delta''_s])(1 + \tan \delta'_s \tan \delta''_s)}{1 + \tan^2 \delta'_s} \dots \dots \dots (15)$$

$$\tan \delta_p = \tan (\delta'_s - \delta''_s + \delta_s) \dots \dots \dots (16)$$

The various angles are defined by the following:

$$\tan \delta_s = \frac{1}{\omega C_2 R_2} = \text{power factor (approximate) of air capacitor } C_2,$$

$$\tan \delta'_s = \frac{1}{\omega C'_1 R'_1} + \frac{1}{\omega C'_1 R'_s} - \frac{K}{\omega C'_1 R'_4}$$

$$\tan \delta''_s = \frac{1}{\omega C''_1 R''_1} + \frac{1}{\omega C''_1 R''_s} - \frac{K}{\omega C''_1 R'_4}$$

$$\frac{1}{\omega C'_1 R'_1} \quad \text{power factor (approximate) of capacitor } C_1 \text{ at first balance, and}$$

$$\frac{1}{\omega C''_1 R''_s} = \text{power factor (approximate) of capacitor } C_1 \text{ at second balance.}$$

As before  $R_1$  and  $R_4$  must be large in comparison to  $\frac{1}{\omega C_1}$  in order that the correction terms be negligible in comparison to unity.

#### VI. Susceptance Variation Method:

In constructing the calibration curve of the parallel-plate capacitor  $C_2$ , the capacitance at any spacing  $S$  is assumed to consist of the "geometric" capacitance (equivalent to  $0.2249 A/S$ ), a constant fringing capacitance  $C_f$ , and an increment correction capacitance  $C_e$  which is the result of lack of linearity in the screw thread, eccentricity and tilt of plates and actual variation in  $C_f$ . At the spacing  $S = 0.100$  in., in accordance with the definition in Section 28 (c),  $C_e = 0$ , and  $C_f$  is thereby determined as the difference between the measured capacitance and the calculated capacitance for that spacing. This value of  $C_f$  is then subtracted from the measured capacitance at each spacing. Let  $S$  now represent the spacing equal to the thickness of the specimen dielectric and  $C'_2$  the equivalent calibration capacitance at that spacing, adjusted for  $C_f$ .  $C_r$  is

the equivalent calibration capacitance, adjusted for  $C_f$  at the spacing which again restores resonance after the removal of  $C_x$ . It is evident that:

$$C_r = C_x + C'_2 - 0.2249 \frac{A}{S}$$

from which Eq. 21 (Section 28 (h)) was derived.

If the specimen has an area  $A_x$ , smaller than  $A$ , it is likewise evident that:

$$C_r = C_x + C''_2 - 0.2249 \frac{A_x}{S}$$

from which Eq. 23 (Section 28 (j)) was derived.

APPENDIX III<sup>16</sup>

## CELLS USED FOR MEASURING POWER FACTOR AND DIELECTRIC CONSTANT OF INSULATING LIQUIDS

A1. *Design of Cell.*—A cell for the purpose of measuring power factor and dielectric constant of electrical insulating liquids should meet the following general requirements:

(a) The design of the cell shall be such as to facilitate easy and thorough cleaning. The design, also, shall conveniently permit the use of the cell in a suitable temperature bath and means shall be provided for measuring the temperature of both electrodes.

(b) The materials used in constructing the cell shall be capable of satisfactorily withstanding temperatures up to 130 C. and the alignment of the electrodes shall not be influenced by this temperature.

platinum. In general, plated surfaces have been found less satisfactory than solid metal electrodes for measuring low power factor liquids. Plated surfaces that may be satisfactory for testing liquids with low acidity and for short periods of time are gold, platinum, nickel, chromium over nickel, or rhodium.

(d) A guard electrode shall be provided which adequately shields the measuring electrode. A shielded wire or coaxial cable shall be used to connect the guard and measuring electrode either directly or by a plug to the bridge.

(e) The solid insulation used to support the guard electrode relative to the measuring electrode shall not extend into the portion of the sample being tested.

(f) The insulating materials used in constructing the cell shall not absorb or be adversely affected by the test liquids, or cleaning solvents. Their power factor is preferably low, particularly

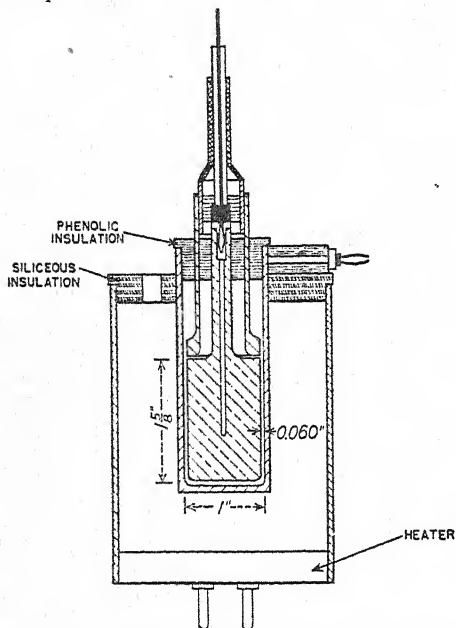


FIG. 17.—Power Factor Cell With Heater.

(c) The electrodes or their surfaces shall be made of a metal capable of resisting attack by mild acids found in certain insulating liquids such as petroleum oil, particularly after prolonged exposure at elevated temperatures. Metals which have been found satisfactory from this standpoint are gold, nickel, monel, and

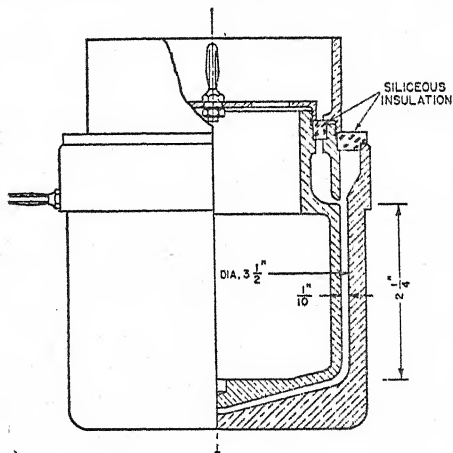


FIG. 18.—Power Factor Cell.

that between the guard and measuring electrodes, otherwise difficulty may be experienced in balancing the guard circuits at elevated temperatures. Insulating materials that have proven satisfactory are pyrex glass, quartz, steatite, and the better phenolic resin compounds. Thermoplastic materials such as hard rubber and polystyrene, although having good electrical properties, are not suitable as they soften below 130 C. Insulating materials of the molded mica-dust type have been found to absorb solvents and therefore may not be satisfactory in the measurements of low power factor liquids. However, they are excellent materials for supporting the cell in the temperature bath.

<sup>16</sup> This appendix was added editorially in January, 1947. It covers requirements for test cells for power factor and dielectric constant which formerly appeared as a tentative revision of the Standard Methods of Testing Electrical Insulating Oils (A.S.T.M. Designation: D 117-43) and for the cell shown in Fig. 19, which is identical with that shown in Appendix III of the Standard Methods of Testing Solid Insulating and Treating Compounds Used for Electrical Insulation (A.S.T.M. Designation: D 176). These cells are referred to in Section 11 (d) of these Methods D 150.

(g) In designing the cell the distance across the surface of the oil and across the solid insulating material, between the guard and the measuring electrode, shall be great enough adequately to withstand the testing potential used. Leakage across these paths has been found to produce an unsteady bridge balance on some bridges.

(h) A cell and heater bath design that is available and is used successfully by several testing laboratories is shown in Fig. 17. Its volume is 8 ml. The voltage limit is 1000 v. and the capacitance with oil having a dielectric constant of 2.2 is about 70 micro-microfarads.

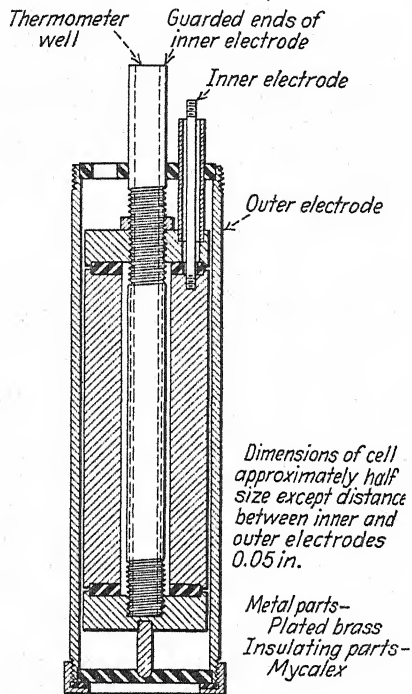


FIG. 19—Measuring Cell for Resistivity, Power Factor, and Dielectric Constant Determinations.

(i) For routine work, cells on the same design principles, with particular attention to quickness of assembly and suitable for higher voltages may be desirable. Several laboratories use cell designs similar to Fig. 18 which are commercially available. This design requires about 90 ml. of oil and is suitable for use up to 3 kv., the capacitance with oil being about 180 micro-microfarads.

(j) A conductivity cell that has been developed for the purpose of measuring the volume resistance, as well as other electrical constants, of solid filling and treating compounds and oils is shown in Fig. 19.<sup>17</sup>

A2. Cleaning Procedure.—(a) The following

is a suggested method for cleaning the cells shown in Figs. 17 and 18:

(1) Fill the test cell with technical grade carbon tetrachloride, insert the inner electrode and bring to a boil on a hot plate. Empty, and repeat this procedure until the carbon tetrachloride poured off is clean.

(2) Wash the inner and outer electrodes with lava soap and water to remove films left by the carbon tetrachloride, taking precautions not to lay the inner electrode on any surface. The use of the lava soap will keep the cell surfaces polished. Rinse all surfaces with distilled water. Place the cell in an oven and maintain at 115 C. for at least 1 hr. prior to taking measurements.

(b) A suggested method of cleaning the cell shown in Fig. 19 is to remove the bottom retaining ring and to hang the cell in an oven by a hook fastened through a hole drilled in the protruding stem. Upon heating, the outer cylinder will slide away from the inner electrode and both parts will drain fairly clean of the compound. In order to speed up this cleaning, the flame of a bunsen burner may be applied directly to the cell after removing the cap and suspending the cell by the protruding stem. Further application of the flame to the cylinder and inner electrode after they separate will rapidly remove most of the compound and the final cleaning of each part can be accomplished by suitable solvents.

A3. Filling the Cell.—(a) Remove the cell from the oven and rinse the electrodes with some of the sample oil to be tested which has been heated to approximately the test cell temperature. Fill the cell with the sample oil which has been heated to approximately the test cell temperature. Permit the assembly to cool to approximately the temperature at which the measurements are to be taken and place in a temperature-controlled device. Remove bolts and handles and connect the cell to the measuring bridge using the guard screen. Measure when the temperature is within plus minus 0.5 C. of the required value.

(b) Remove the inner electrode, pour off this sample and refill with a second taking the same precautions as were observed in the first filling and make another measurement. If these two values disagree by more than  $0.0001 \pm 10$  per cent of the measured power factor, refill and make a third measurement.

(c) If the third value does not agree with either the first or second measured values to within  $0.0001 \pm 10$  per cent of the measured power factor, reclean the cell and repeat the procedure until two successive readings are obtained which do agree to within  $0.0001 \pm 10$  per cent of the power factor.

<sup>17</sup> This figure and instructions for operating this cell appear in Appendix III of the Standard Methods of Testing Solid Filling and Treating Compounds Used for Electrical Insulation (A.S.T.M. Designation: D 176), see p. 106.



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## *Tentative Method of Test for*

# POWER FACTOR AND DIELECTRIC CONSTANT PARALLEL WITH LAMINATIONS OF LAMINATED SHEET AND PLATE INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 669 - 42 T

ISSUED, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers the procedure for determining the power factor and dielectric constant of stiff laminated sheet and plate insulating materials in a direction parallel with the laminations (Note). The method primarily includes information covering the preparation of the specimen and details concerning the procedure required to make measurements parallel with the laminations for this particular type of material. The apparatus and general test procedure shall be in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup>

NOTE.—It has long been recognized that the power factor and dielectric constant of laminated insulation as measured in a direction parallel

with the laminations, are not of the same magnitude as those taken perpendicular with the laminations.

### Specimen Holder

2. The specimens shall be clamped in a suitable holder consisting of a frame constructed from  $\frac{3}{8}$ -in. square metal rod of a size suitable for holding the test specimens and clamping them tightly. A slot 0.260 in. in width and  $\frac{1}{16}$  in. in depth shall be milled in the inner opposite parallel sides of the supporting frame to hold the specimens in a parallel position, suitable for test. Threaded studs shall be inserted in the open ends of the slotted parallel bars to receive the clamping bar which shall be drawn down tightly on the specimen pile-up by means of nuts (see Fig. 1).

### Electrodes

3. Thin tin or lead foil electrodes shall be used. These shall be from 2 to 4 in. in diameter depending upon the frequency at which the test is made, as well as the type of circuits (see Sections

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1942.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

22 to 28 of Tentative Methods D 150).<sup>3</sup>

A very thin coating of petrolatum shall be used as an adhesive for the foil electrodes. A smoothing pressure sufficient to eliminate all wrinkles shall be applied by use of a suitable roller or piece of soft cloth, making sure that there is no air gap between the foil and specimens due to the unevenness of the specimen pile-up. The electrodes shall be accurately

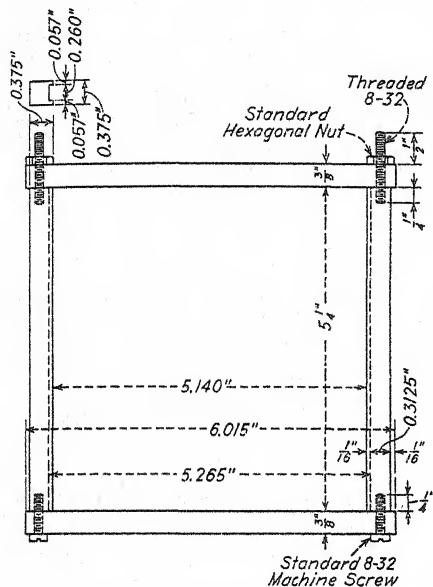


FIG. 1.—Specimen Holder.

centered with respect to each other and at least approximately centered with respect to the specimen.

## Test Specimens

4. (a) Test specimens from sheets of any thickness may be tested. Strips shall be cut 0.250 in. in width plus 0.005 in. minus 0.000 in., by the thickness of the sheet, and  $5.25 \pm 0.015$  in. in length, in sufficient quantity to make a pile-up approximately 5.25 in. in height, to form the test specimen. The strips shall be mounted in the specimen holder with the uncut surfaces placed together and the sawed edges exposed, thus placing

the laminations in a parallel direction with the electrostatic field created by the electrodes placed on each side of the strip pile-up. When thick sheets are tested it shall be permissible to reduce the thickness of the outside strip to permit mounting in the frame. The surfaces shall be smooth and free from irregularities. It is essential that all the burrs on the cut strips be removed to insure that no air gaps are created between adjacent strips or between strips and foil electrodes. Sanding of irregular surfaces to eliminate air gaps between strips in the pile-up shall be permissible provided the total resin content or character of the sheet is not changed thereby. A very smooth surface for mounting the electrodes may be obtained by taking a light cut with a milling machine or lathe from both surfaces of the pile-up after the specimens have been mounted in the frame.

(b) The strips, after being mounted in the frame, shall be placed under pressure by drawing down the two nuts on the crossbar. Sufficient pile-up shall be used so that the crossbar is not brought in contact with the two parallel bar supports, thus insuring that the specimen strips are under compression. After the specimen pile-up has been assembled into the frame, it is well to lay it on a flat surface so that the strips and not the frame are resting on this surface. This ensures that all surfaces on one side will be in one plane while being clamped by the crossbar.

(c) The capacitance of the specimen shall be, preferably, not less than 50  $\mu\text{mf}$ . Use of smaller specimens increases the effects of stray capacitances and decreases the precision of measurements.

### Preconditioning Specimens

5. Before mounting in the test frame the specimen shall be preconditioned at a temperature of  $50 \pm 3$  C. for a period

of 48 hr. After removal from the oven, the specimen shall be allowed to cool for a period of at least 1 hr. in a desiccator in order to be certain that it has reached room temperature. Other conditioning periods as agreed upon between the seller and the purchaser may be used.

### Procedure

6. (a) The power factor, dielectric constant, and loss factor shall be determined in accordance with the procedure described in Sections 15 to 28 of Tentative Methods D 150,<sup>3</sup> except that tests at other than room temperature shall be made as described in Section 14 (a) and (b) of Tentative Methods D 150.<sup>3</sup>

(b) The conditioned specimens shall be mounted in the frame and tested as soon as practicable and preferably within 10 min. after removal from the desiccator.

(c) Tests shall be made on specimens cut in the lengthwise direction of the sheet, also on specimens cut in the crosswise direction. Where the machine direction of paper-base sheets or the warp direction of fabric base sheets is known it shall constitute the lengthwise direction. Otherwise the longer direction of a sheet shall be considered the lengthwise direction.

NOTE.—When the sheet has the same length and width, one dimension shall arbitrarily be designated as the length.

(d) For the accurate measurement of thickness for the dielectric constant calculation, it is recommended that

when the surfaces are not milled or lathe-turned after assembly, the individual strips shall be measured for thickness in a direction parallel with the laminations, and an average of these measurements taken as the true thickness.

### Report

7. The report shall include the following:

(1) A description of the material: that is, the name, grade, and color, and the name of the manufacturer,

(2) The following test conditions: The frequency in cycles (or kilocycles) per second, temperature of test in degrees Centigrade, percentage relative humidity of the atmosphere, conditioning of the specimen (hours at temperature and humidity), the type of electrodes, and the voltage gradient in the dielectric while under test,

(3) The following values for the test specimen: The capacitance of each specimen comprised of strips cut lengthwise and of strips cut crosswise of the sheet, expressed in micro-microfarads, effective area of the specimen electrodes for the lengthwise and crosswise cut specimens, the average thickness of the specimen between the electrodes for the lengthwise and crosswise cut specimens,

(4) The power factor, dielectric constant, and loss factor of each specimen tested for determinations on specimens cut both in the lengthwise direction and in the crosswise direction, and

(5) Method of measurement.

# *Tentative Methods of*

## SAMPLING AND TESTING UNTREATED PAPER USED IN ELECTRICAL INSULATION<sup>1</sup>



**A.S.T.M. Designation: D 202 - 46 T**

ISSUED, 1924; REVISED, 1925, 1926, 1927, 1928, 1929, 1930, 1932, 1933,  
1934, 1936, 1938, 1939, 1940, 1941, 1944, 1945, JUNE 1946, NOVEMBER 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover the procedures for sampling and testing untreated paper to be used as an electrical insulator or as a constituent of a composite material used for electrical insulating purposes.

### Sampling

2. (a) For batches consisting of rolls 15 in. or more in width, a sample at least 5 sq. ft. in area cut across the entire width of the roll shall be taken from every tenth roll of the batch, chosen in such a manner as to well represent the entire batch. In case the paper is available in rolls less than 15 in. in width, the length of the sample to be taken may be such that the area is less than 5 sq. ft. but shall be at least 4 ft. in length.

Where paper is in ribbon or pad form and in dowels; that is, a number of pads assembled on a dowel stick in the same order as slit from the parent roll, the

sample should consist of specimens taken from the pads so that the entire width of the parent roll is represented. A minimum number of pads shall be sampled, the total width of which shall be equal to at least ten per cent of the width of each dowel. A minimum number of 4 pads from each dowel shall be sampled.

For batches in sheet form, sample sheets totaling at least 5 sq. ft. in area shall be taken from each tenth bundle or package chosen in such a manner as to well represent the entire batch.

In the case of either type of package no less than 5, nor more than 20 units shall be sampled, taken as follows:

Total Units in Shipment	No. of Units Selected
Less than 100.....	5
100 to 399.....	5 per cent
400 and over.....	20

NOTE.—It is recommended that in the case of paper in roll form, several turns be discarded before the samples are taken.

(b) The tests for physical properties shall be made on each sample insofar as the specimens are of sufficient width. If the samples are to be used for the de-

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Latest revisions accepted by the Society at annual meeting, June, 1946, and through the Administrative Committee on Standards, November, 1946.

termination of moisture in the batch, extreme care shall be taken to avoid change in moisture content in the samples before they are weighed as is provided for in the moisture method (Section 4).

### Conditioning

3. (a) Samples shall be conditioned in air maintained at a relative humidity between 60 and 65 per cent as measured with a sling psychrometer or its equivalent. The temperature of the air shall be maintained as constant as possible at some temperature between the limits of 20 and 30 C. (68 and 86 F.). The samples should remain in the conditioned air for not less than 4 hr. prior to the tests and should be supported so as to allow a free circulation around each sample.

(b) The following physical tests shall be made in the conditioned atmosphere: thickness, weight, tensile strength, tearing strength, bursting strength, folding endurance, air resistance, absorption, conducting paths, and rate of impregnation.

### MOISTURE CONTENT

#### Procedure

4. Moisture content shall be determined in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (A.S.T.M. Designation: D 644),<sup>3</sup> except that the material shall be sampled in accordance with Section 2.

### THICKNESS

#### Test Specimens

5. (a) Test specimens shall be taken from the original samples obtained in accordance with Section 2 (a). The test specimens shall be conditioned as prescribed in Section 3.

(b) For papers over 0.002 in. in thick-

ness, the specimen shall be a single sheet.

(c) For papers 0.002 in. and under in thickness, the specimen shall consist of a stack of ten sheets (Note).

NOTE.—A multiple-sheet stack is used for papers 2 mils and under in thickness because of the greater reliability of micrometer measurements made at the resulting larger micrometer surface separations. It should be borne in mind that the thickness of a ten-sheet stack of paper bears no constant relation to the thickness of a single sheet. It is also pointed out that variations in single sheet thickness are largely hidden in multiple-sheet stack measurements.

#### Procedure

6. (a) The thickness shall be determined in accordance with method A or method C of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials.<sup>3</sup>

(b) At least five measurements of thickness shall be taken at regular intervals across the entire width of each specimen, preferably in a line which is at right angles to the machine direction.

(c) When using multiple-sheet stacks for thickness measurements, the micrometer foot shall not be closer than 0.75 in. from any folded edge of the stack. The stack thickness divided by the number of sheets in the stack shall be reported as the thickness of the specimen.

#### Report

7. The average, maximum, and minimum thicknesses obtained on each specimen shall be reported. In cases where the multiple-sheet stack method is used, a statement shall be made to that effect.

### WEIGHT

#### Procedure

8. The determination of weight shall be made in accordance with the Standard Method of Test for Basis Weight of Paper and Paper Products (A.S.T.M. Designation: D 646)<sup>3</sup>, except for sam-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



pling and conditioning of the sample which shall be carried out in accordance with Sections 2 and 3 of Methods D 202. The results shall be reported as weight in grams per square meter.

### APPARENT DENSITY

#### Procedure

9. The apparent density shall be calculated from the thickness (Sections 5 to 7), area, and weight (Section 8) results obtained on conditioned specimens (Section 3), and reported as grams per cubic centimeter.

NOTE.—Apparent density may be calculated as follows:

$$D = \frac{\text{grams per square meter}}{\text{thickness in inches}} \times 0.0000394$$

$$D = \frac{\text{weight in grams}}{\text{volume in cubic inches}} \times 0.061$$

$$D = \frac{\text{weight of 25 by 40 in. — 500 sheet ream in pounds}}{\text{thickness in inches}} \times 0.0000554$$

$$D = \frac{\text{weight of a 7-13/16 in. square sample in grams}}{\text{thickness in mils}}$$

where:

$D$  = apparent density in grams per cubic centimeter.

### TENSILE STRENGTH

#### Apparatus

10. (a) A testing machine of the dead-weight pendulum type suitably designed for testing paper shall be used. The machine shall preferably be power driven.

(b) The capacity of the machine shall not exceed 113 kg. (250 lb.).

(c) The machine shall be graduated to read 1 kg. or 1 lb. or less per scale division for testing paper breaking at 22.7 kg. (50 lb.) or over, and to read 0.5 kg. or 0.5 lb. or less per scale division for testing paper breaking under 22.7 kg. (50 lb.).

### Test Specimens

11. The test specimens shall be cut from each of the samples selected in accordance with Section 2 (a). The specimens shall not exceed 2.54 cm. (1 in.) in width and shall be 25.4 cm. (10 in.) in length with clean-cut edges. At least ten specimens shall be obtained in the machine direction and, if practicable, ten in the cross-machine direction. The specimens shall be conditioned as prescribed in Section 3.

#### Procedure

12. (a) The ratio of the clearance distance between jaws to the width of the specimen shall be not less than 5 to 1 nor more than 10 to 1.

(b) The rate of travel of the movable jaw shall be constant. It shall preferably be 30.5 cm. (12 in.) per min., but it may be within the limits of 28 cm. (11 in.) and 33 cm. (13 in.) per min. provided it is constant.

(c) All readings obtained when the specimen breaks at or in the jaws shall be rejected.

#### Report

13. The report shall include the following:

(1) The tensile strength in kilograms or pounds of each machine-direction specimen and of each cross-machine-direction specimen, reported separately.

(2) The width of each specimen in centimeters or inches, and also the average thickness.

(3) The maximum, minimum, and average breaking load shall be reported for the tests on the machine-direction specimens and for the cross-machine-direction specimens.

### TEARING STRENGTH

#### Procedure

14. Tearing strength shall be determined in accordance with the Standard



Method of Test for Internal Tearing Resistance of Paper (A.S.T.M. Designation: D 689),<sup>3</sup> except that the material shall be sampled and conditioned in accordance with Sections 2 and 3.

#### BURSTING STRENGTH

##### Apparatus

15. The testing machine shall have a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber shall be filled with glycerol or other suitable pressure medium and shall contain no air spaces. The test specimen shall be held in position over the diaphragm in a clamp having a circular hole approximately 6.44 sq. cm. (1 sq. in.) in area so that the diaphragm will force the paper into the hole when pressure is applied under the diaphragm. The pressure exerted on the diaphragm shall be indicated on a dial, graduated to at least 0.23 kg. (0.5 lb.) for papers having bursting strengths 18 kg. (40 lb.) or under. A dial graduated to 0.45 kg. (1 lb.) may be used in testing papers which have higher bursting strengths. The machine may be either hand-operated or power-driven, the latter being preferable.

##### Test Specimen

16. If practicable, the specimen shall be so cut from the sample as to permit ten bursting tests on a line across the sheet or roll. The specimens shall be conditioned as prescribed in Section 3.

##### Procedure

17. Ten bursts shall be made, five with one side of the specimen uppermost and five with the other side uppermost. The testing machine shall be operated at a uniform speed of 120 rpm. until the specimen bursts.

##### Report

18. The report shall include the

average, maximum, and minimum results obtained.

#### FOLDING ENDURANCE

##### Procedure

19. Folding endurance shall be determined in accordance with method B in the Standard Methods of Test for Folding Endurance of Paper (A.S.T.M. Designation: D 643),<sup>3</sup> except that the material shall be sampled and conditioned in accordance with Sections 2 and 3.

#### ABSORPTION

##### (Rise of Water)

##### Apparatus

20. The apparatus shall consist of a suitable container, and a support for the test specimens and scale as shown in Fig. 1.

##### Test Specimens

21. Ten test specimens 2.5 cm. (1 in.) in width and at least 12.5 cm. (5 in.) in length shall be cut from the samples obtained in accordance with Section 2 (a), five being cut parallel with the machine direction of the paper and five cut parallel with the cross-machine-direction of the paper. The specimens shall be conditioned as prescribed in Section 3.

##### Procedure

22. The specimens shall be suspended vertically with one end dipping 3.2 mm. (0.125 in.) in distilled water at room temperature, and after 5 min. the rise of the water in the specimen above the level of the water shall be noted. The rise may be measured by reading directly from the scale the height of absorption.

##### Report

23. The absorption shall be reported as the rise of water in millimeters to the nearest 3 mm. or in inches to the nearest  $\frac{1}{8}$  in. The average and individual re-

sults for both the machine direction and the cross-machine direction of the paper shall be reported.

#### AIR RESISTANCE

##### Apparatus

24. (a) The apparatus shall consist of two aluminum open-top cylinders, one of which is inverted and slides into the other which is fixed. The movable cylinder shall be provided with a circular

surface of the fixed cylinder to act as guide tracks for the movable cylinder. The movable cylinder shall be graduated in units of 50 ml. and shall have a total range of 350 ml. It shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 7.62 cm. (3.00 in.) and an internal diameter of 7.35 cm. (2.90 in.). It shall weigh, including the flat ring clamp and the two knurled nuts,  $567 \pm 0.5$  g. ( $20 \pm 0.018$  oz.). The

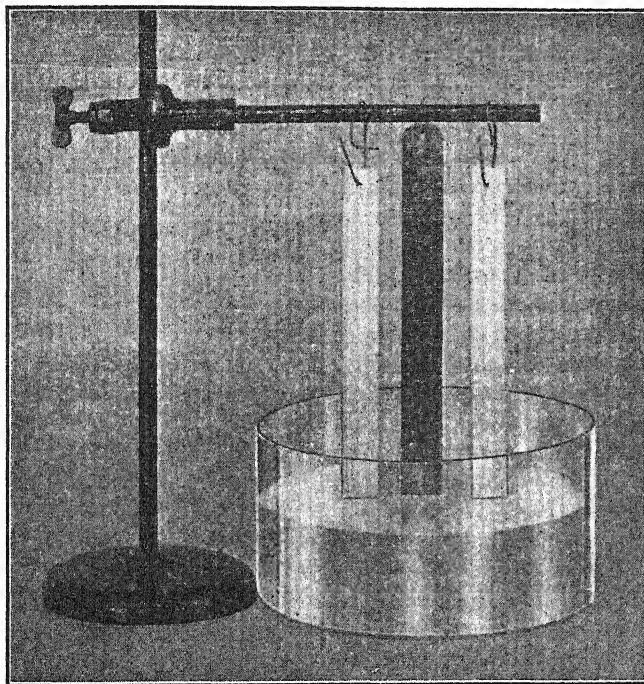


FIG. 1.—Apparatus for Absorption Test.

aperture in the closed end and a flat ring clamp for holding the paper specimen across this aperture.

(b) The fixed cylinder shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 8.6 cm. (3.38 in.) and an internal diameter of 8.25 cm. (3.25 in.). Four slender bars, each 18.5 cm. (7.3 in.) in length, 3 mm. (0.12 in.) in width, and approximately 1.5 mm. (0.06 in.) in thickness shall be mounted vertically and equidistantly on the inner

movable cylinder and the flat ring clamp shall have a concentric circular aperture of 6.44 sq. cm. (1.00 sq. in.) in area. When this aperture is too large for the specimen, a similar movable cylinder and flat ring clamp having a concentric circular aperture of 1.61 sq. cm. (0.25 sq. in.) in area shall be used.

NOTE.—Results obtained with apertures of different areas are not directly proportional to the areas of the apertures. The proportionality factor must be determined by experiment.

### Test Specimens

25. The test specimens shall be cut from the original samples obtained in accordance with Section 2 (a). They shall be not less than 3.5 cm. (1.36 in.) nor more than 5.1 cm. (2.0 in.) in width, and shall be as long as the width of the original roll of paper. If the specimens are selected from pads of tape they shall be not less than 2.1 cm. (0.81 in.) nor more than 5.1 cm. (2.0 in.) in width and at least 30 cm. (12 in.) in length. There shall be as many specimens as there are original samples. The specimens shall be conditioned as prescribed in Section 3.

### Procedure

26. The fixed cylinder shall be placed on a rigid support so that its sides are vertical. A lubricating oil (Note 1) having a viscosity of 60 to 70 sec. Saybolt Universal at 37.8 C. (100 F.) shall be placed in it to a depth of 12.7 cm. (5 in.). The specimen (one thickness only) shall be secured tightly under the clamp (Note 2) on the movable cylinder, completely covering the aperture. The movable cylinder shall then be floated on the lubricating oil (Note 3). The time required for the displacement of a certain amount of air shall be noted with a stop watch. If possible, the amount of air displaced should be such that the time of displacement is not less than 20 sec. The apparatus with its content of lubricating oil shall be at the temperature of the conditioning room (Section 3) when the readings are taken. The time in seconds required for the displacement of 100 ml. (6.1 cu. in.) of air through a circular area (one side only) of 6.44 sq. cm. (1 sq. in.) of the specimen shall be considered as the air resistance of the paper.

NOTE 1.—Oil is used in preference to distilled water because it does not corrode aluminum.

NOTE 2.—The proper procedure for clamping the specimen or tinfoil is to turn both knurled

nuts down onto the clamp simultaneously. If only one nut at a time is turned down, the clamp will not fit flat on the specimen and will consequently have an avoidable leak. The clamp shall be tested for leakage by substituting a piece of tinfoil 0.05 mm. (0.002 in.) in thickness for the paper specimen and testing in the manner described in Section 36. When so tested, the leakage shall not exceed the rate of 50 ml. in 5 hr.

NOTE 3.—Precautions should be taken to avoid subjecting the apparatus to vibration as this condition would increase the rate of air displacement.

### Report

27. The report shall include the following:

(1) The number of seconds required for the displacement of 100 ml. (6.1 cu. in.) of air,

(2) The area of paper through which the air was displaced,

(3) The thickness of the paper specimen as obtained under Sections 5 to 7, and

(4) The room temperature.

NOTE.—The following values show the probable reproducibility of results obtainable in the air-resistance tests:

Air Resistance	Reproducibility of Results
40 sec.....	±5 per cent
100 sec.....	±6 per cent
200 sec.....	±8 per cent
300 sec.....	±10 per cent

### ASH

### Procedure

28. The determination of ash content shall be made in accordance with the Standard Method of Test for Ash Content of Paper and Paper Products (A.S. T.M. Designation: D 586) of the American Society for Testing Materials.<sup>3</sup>

### ACIDITY OR ALKALINITY

### Apparatus

29. The apparatus shall consist of the following:

(a) *Bath*.—A hot water bath.

(b) *Motor and Stirrer.*—A motor with a stirrer constructed as shown in Fig. 2. The stirrer shall be made of brass, chromium plated to render it acid and alkali resistant.

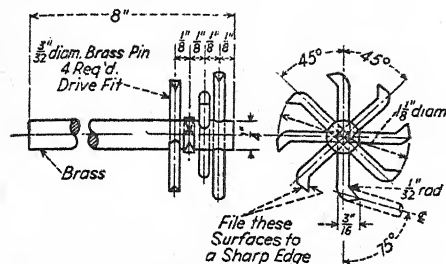


FIG. 2.—Stirrer.

(c) *Flasks.*—Wide mouth acid-resistant and alkali-resistant glass<sup>4</sup> 250 ml. Erlenmeyer flasks.

(d) *Thermometers.*—Thermometers having a range of from 50 to 100 C. and graduated in 1 C. intervals.

(e) *Burette.*—A 10-ml. burette graduated to 0.05 ml.

(f) *Electric Hot Plate.*

(g) *Suction Filtering Apparatus.*

## Reagents

30. (a) *Distilled Water.*—Adjust the distilled water used in making the extractions to a pH of 7.0 to 8.0 when free of carbon dioxide. Carbon dioxide can be eliminated by boiling or by bubbling nitrogen through the solution during the pH measurement or titration. The following method of adjusting the pH of water to be used in making extractions is recommended: Carry out a titration on 100 ml. of the water according to the prescribed procedure under "blank titration" Section 32 (d). If more than 0.10 ml. of 0.005 *N* alkali solution is required to obtain an end point with phenolphthalein, adjust the pH of the water to be used by adding the calculated amount of alkali so that the blank titration on 100 ml. is 0.10 ml. or less of 0.005 *N*

NaOH (that is, the water used should be neutral or only slightly acid to the phenolphthalein indicator).

(b) *Standard Sodium Hydroxide Solution (Approximately 0.005 *N*).*—To standardize, prepare 250 ml. of 0.005 *N* potassium acid phthalate solution by dissolving 0.2552 g. of the dried salt (National Bureau of Standards) in water and making up to the mark in a 250-ml. volumetric flask at 20 C. (Do not dry the salt at a temperature above 125 C.) Pipette 25 ml. of this solution into a 250-ml. flask. Add 25 ml. of water and 5 drops of phenolphthalein solution. Pass nitrogen through the solution for 10 min. Titrate in a closed system with the standard NaOH solution to the first permanent pink shade. If preferred, the potassium acid phthalate solution may be heated to boiling and titrated immediately, taking care that the temperature does not fall below 80 C. during the titration. Run three samples in the above way at each standardization of the NaOH. Determine a blank on the same volume of distilled water and phenolphthalein and deduct from the titration obtained above. Calculate the normality by the following formula:

$$\text{Normality of NaOH} = \frac{25 \times 0.005}{\text{milliliters of NaOH required}}$$

(c) *Sulfuric Acid (Approximately 0.005 *N*).*—Determine the alkali equivalent of the acid as follows: Transfer 10 ml. of the acid to a 250-ml. Erlenmeyer flask and dilute with 100 ml. of distilled water. Titrate in a closed system or at the boiling point with the standard NaOH solution as described in Paragraph (b) for the standardization of the NaOH solution. Determine a blank on the same volume of distilled water and phenolphthalein and deduct from the

<sup>4</sup> Pyrex glass has been found very satisfactory for this purpose.

titration obtained above. Calculate the NaOH equivalent of the acid as follows:

$$E = \frac{A}{B}$$

where:

$E$  = NaOH equivalent (in milliliters) to 1 ml. of  $H_2SO_4$ ,

$A$  = milliliters of NaOH required (corrected), and

$B$  = milliliters of  $H_2SO_4$  taken.

(d) *Phenolphthalein Solution*. — Dissolve 0.5 g. of phenolphthalein in 100 g. of c.p. ethyl alcohol (95 per cent).

### Test Specimen

31. From the samples obtained in accordance with Section 2 (a), a composite test specimen weighing at least 5 g. shall be cut into small pieces, approximately 1-cm. (0.4-in.) square. The specimen shall be thoroughly mixed and, during preparation, any contamination by handling shall be avoided.

### Procedure

32. (a) A 1-g. portion of the composite specimen shall be placed in the 250-ml. Erlenmeyer flask and 100 ml. of boiling distilled water added. The flask shall then be clamped in position in a boiling water bath so that at least one-half of the flask is immersed in the water bath. During the stirring, the temperature of the contents of the flask shall remain at least as high as 95 C. The stirrer shall be mounted so that the blades are within 1 cm. (0.4 in.) of the bottom of the flask. The assembled extraction apparatus is shown in Fig. 3. The stirrer shall be driven at a speed of 4000 to 5000 rpm. for 5 min. At the end of this period the specimen should have been thoroughly pulped. For paper unusually difficult to pulp, the period of stirring shall be increased to 10 min.

(b) Immediately after the specimen has been pulped, the contents of the

flask shall be filtered rapidly into another 250-ml. Erlenmeyer flask without washing.

NOTE.—It is important that the filtration of the extract be accomplished as promptly as possible after disintegration. To insure rapid filtration, filter, using suction, through a No. 42 Whatman paper (which has been washed twice with 100-ml. portions of hot distilled water) on a Büchner funnel. Use a suction bell jar and collect the filtrate in the Erlenmeyer flask.

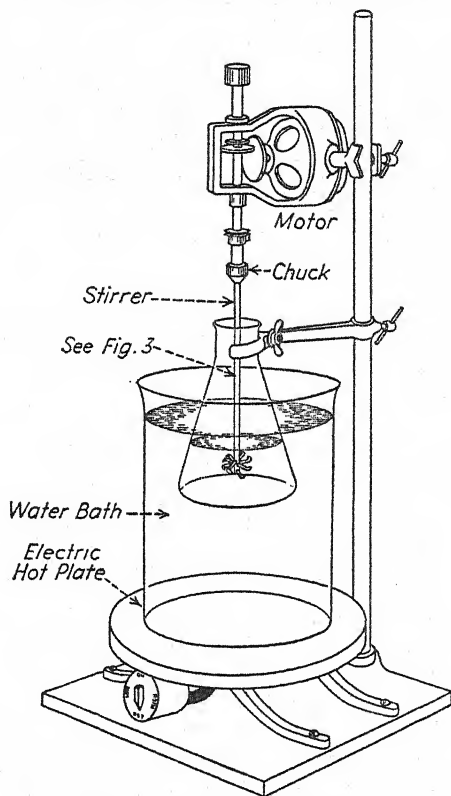


FIG. 3.—Extraction Apparatus.

(c) The filtered extract shall be titrated as described in Section 30 (b) for standardizing the alkali solution. If the extract is acid, it shall be titrated with 0.005  $N$  NaOH, using 5 drops of phenolphthalein solution as indicator. The appearance of a definite pink color indicates the end point. If the extract is alkaline, a small excess of 0.005  $N$

H<sub>2</sub>SO<sub>4</sub> shall be added and the titration then completed with 0.005 *N* NaOH.

(d) *Blank*.—The titration obtained in Paragraph (c) shall be corrected for the blank error. This correction shall be determined by running a blank in parallel with the actual determination using a volume of pure distilled water equal to that of the extract at the end point. If the solution is acid, this blank correction shall be subtracted from the volume of alkali used; if alkaline, the correction shall be added.

(e) At least two specimens shall be tested. If the titrations on duplicate specimens do not agree within 0.10 ml., the determinations shall be repeated.

#### Calculations and Report

33. (a) The acidity or alkalinity shall be calculated as follows:

$$M = \frac{a \times N}{W}$$

where:

*M* = milliequivalents of acid or alkali per gram of paper,

*a* = milliliters of NaOH required (corrected),

*N* = normality of the NaOH, and

*W* = weight of sample in grams.

(b) The results shall be reported as the number of milliequivalents of acid per gram if the extract is acid, or as the number of milliequivalents of alkali per gram if the extract is alkaline, the number of milliequivalents being based on the weight of the air-dry sample.

#### CONDUCTING PATHS

##### Purpose

34. The test for conducting paths in untreated insulating paper consists of the determination of the number of electrical breakdowns in specified areas of the paper when it is subjected to voltages somewhat lower than those obtained as the dielectric strength values when the

paper is tested in accordance with the short-time test described in the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>3</sup> Three test methods are covered, for papers within each of the three thickness ranges as follows:

*Method A*.—For papers 0.038 mm. (0.0015 in.) and under in thickness,

*Method B*.—For papers over 0.038 mm. (0.0015 in.) and under 0.76 mm (0.030 in.) in thickness, and

*Method C*.—For papers 0.76 mm. (0.030 in.) and over in thickness.

#### Test Specimens

35. The test specimens shall be of relatively large area, such as a pad of tape, a sheet from each sample of paper, or a portion of a paper roll.

*Method A. For Papers 0.038 mm. (0.0015 in.) and Under in Thickness*

#### Apparatus

36. The apparatus shall consist of the following:

(a) *Electrodes*.—Two electrodes consisting of a smoothly machined cast iron or other metal plate, and a hand operated metal roller with smoothly machined face and fitted with an insulated handle. The plate shall be of convenient size to accommodate the specimens to be tested. The finished dimensions of the roller shall be as follows:

	Centimeters	Inches
Diameter .....	5.1	2.0
Width of face .....	2.5	1.0

NOTE.—A roller with a width of face of 0.95 cm. (0.37 in.) should be used on papers under 1.50 in. in width.

(b) *Receiver*.—A telephone headset with two receivers, each of 1500-ohm resistance connected in series.



(c) *Resistor*.—A resistor of 80,000 ohms.

(d) *Voltage*.—A source of 110 v. Either alternating or direct voltage may be used, but the type chosen should be considered in establishing specific limits, inasmuch as different results will probably be obtained with the two types of voltage.

#### Test Specimen

37. (a) The test specimen shall be of sufficient size to cover the plate electrode and shall be held in place by weights. Several such specimens shall be tested. The paper shall contain not more than 8 per cent of moisture when tested.

(b) It has been found in the testing of thin papers that great care must be taken in handling the specimens. They must not be touched with the hands nor should they be laid upon anything but the plate electrode after removal from the roll.

#### Procedure

38. The test specimen shall be secured flat upon the plate electrode by means of weights at the ends. The plate shall be connected to the grounded pole of the 110-v. circuit. The roller, the telephone receivers, and the 80,000-ohm resistor shall be connected in series in that order, and the remaining terminal of the 80,000-ohm resistor attached to the live pole of the 110-v. circuit. The roller shall be passed once over parallel sections of the test specimen, avoiding overlapping, at a speed of 5 to 20 ft. per min. The clicks heard in the telephone receiver shall be counted, each click being considered a conducting path.

#### Calculation and Report

39. (a) *Calculation*.—The number of conducting paths shall be calculated as follows:

$$\text{Number of conducting paths per square foot} = \frac{B}{A}$$

where:

$B$  = total number of clicks counted on all test specimens, and

$A$  = total number of square feet in the 1-in. sections in all test specimens.

(b) *Report*.—The report shall include the following:

- (1) Number of conducting paths per square foot,
- (2) Type of current used, whether alternating or direct current, and
- (3) Number of square feet of paper tested.

*Method B. For Papers Over 0.038 mm, (0.0015 in.) and Under 0.76 mm, (0.030 in.) in Thickness*

#### Apparatus

40. The apparatus shall consist of the following:

(a) *Electrodes*.—Two electrodes consisting of a smoothly machined cast iron or other metal plate, and a set of smooth brass rollers. The plate dimension parallel to the direction over which the test specimen is moved shall be at least 15.2 cm. (6 in.); the other dimension of the plate shall be at least as great as the width of the test specimen.

The surfaces of the rollers shall be machined and polished to smooth cylinders. The finished dimensions of the brass rollers shall be as follows:

	Centimeters	Inches
Diameter .....	3.8	1.5
Width of face .....	2.5	1.0

The rollers shall be mounted in two parallel rows above the plate electrode and so arranged that each roller may be raised above or lowered onto the plate surface, automatically aligning the roller surfaces with the plate surface. The rollers and plate shall be adequately insulated from each other. The rollers

shall be mounted so that continuity of electrical connection between them and the potential source is assured when they are rotating. The two rows of rollers shall be mounted with their axes 90 deg. to the direction in which the test specimen is moved. The rollers in each row shall be spaced  $1\frac{3}{8}$  in. apart on center lines and the two rows so placed that the midpoints of the rollers in one row are opposite the midpoints of the spaces in

width that its edges extend at least 1 in. beyond the outer edge of the adjacent rollers. The test specimen shall be at least 7.61 m. (25 ft.) in length. The paper shall contain not more than 7 per cent of moisture at the time of test.

#### Procedure

42. The rollers shall be raised, the end of the test specimen inserted, and

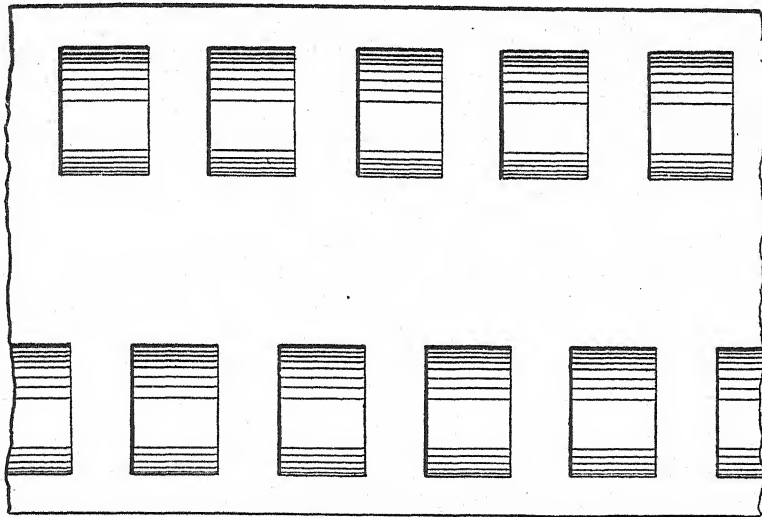


FIG. 4.—Arrangement of Brass Rollers Used in Surveying Medium Thick Papers for Conducting Paths (Method B).

the other row as shown schematically in Fig. 4. The total weight exerted by each roller on the surface of the test specimen shall be not more than 115 g. nor less than 85 g.

(b) *Voltage*.—A source of direct voltage of proper voltage to impress 141 v. per mil on the test specimen, or a transformer with regulating equipment to furnish an alternating potential at a voltage sufficient to impress 100 v. per mil on the test specimen.

#### Test Specimen

41. The test specimen shall be of such

width that its edges extend at least 1 in. beyond the outer edge of the adjacent rollers. The electrodes shall be connected with a potential source of the proper voltage prescribed in Section 40 (b). The specimen shall be drawn between the plate and rollers at a speed of 30 to 60 ft. per min. The number of holes burned through the specimen shall be counted, each burned hole being considered a conducting path.

#### Calculation and Report

43. (a) *Calculation*.—The number of conducting paths shall be calculated as follows:



Number of conducting paths per square foot =  $\frac{H}{A}$       *Method C. For Papers or Boards 0.76 mm. (0.030 in.) and Over in Thickness*

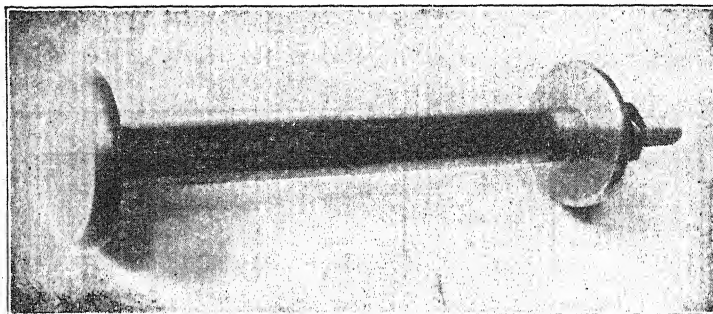
where:

$H$  = total number of holes burned in all specimens, and

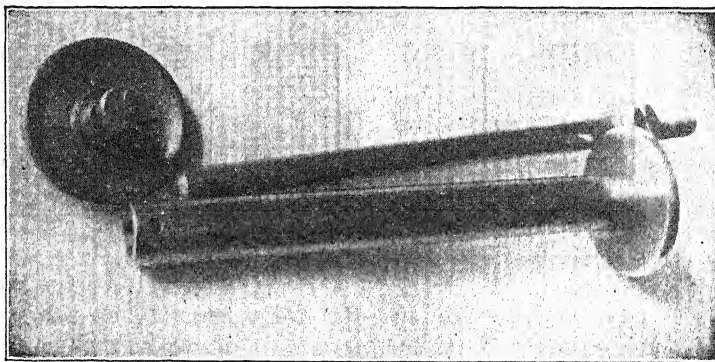
$A$  = total number of square feet contacted with the rollers.

#### Apparatus

44. The apparatus shall consist of the following:



(a) Assembled.



(b) Unassembled.

FIG. 5.—Disk Electrode for Surveying Paper Over 0.76 mm. in Thickness for Conducting Paths (Method C).

(b) *Report*.—The report shall include the following:

- (1) Number of conducting paths per square foot, and
- (2) Number of square feet of paper tested.

(a) *Electrodes*.—Two electrodes consisting of a smoothly machined cast iron or other metal plate and four rows of brass disks. The plate dimension parallel to the direction over which the specimen is moved shall be at least 20.3 cm. (8.0 in.). The other dimension of

the plate shall be at least as great as the width of the test specimen.

The edges of the disk electrodes shall be rounded to a radius of 0.64 cm. (0.25 in.) and the contact surfaces of the disks shall be machined and polished flat and smooth. The finished dimensions of the disk electrodes (see Fig. 5) shall be as follows:

	Centimeters	Inches
Diameter of flat contact surface .....	3.8	1.5
Thickness, min. ....	0.64	0.25

The disk electrodes shall be mounted in four rows parallel to each other and so arranged that each disk may be raised above or lowered onto the plate surface, automatically aligning the disk surfaces with the plate surface. The disks and plate shall be adequately insulated from each other. The four rows of disks shall be mounted at 90 deg. to the direction in which the test specimen is moved. The disks in each of the four rows shall be staggered so that when the test specimen is pulled between the disks and the plate, the entire surface of the test specimen will be contacted twice by the disk electrodes. The total weight exerted by each disk on the surface of the test specimen shall be not more than 700 nor less than 500 g.

(b) *Voltage*.—A source of direct voltage of proper voltage to impress 141 v. per mil on the test specimen, or a transformer with regulating equipment to furnish an alternating voltage at a voltage sufficient to impress 100 v. per mil on the test specimen.

### Test Specimen

45. The test specimen shall be of such width that its edges extend at least 1 in. beyond the outer edge of the adjacent disks. The test specimen shall contain not more than 7 per cent of moisture at the time of test.

### Procedure

46. The disks shall be raised, the test specimen placed on the plate electrode, and the disks lowered onto the paper surface. The electrodes shall be connected with a potential source of the proper voltage prescribed in Section 44 (b). The specimen shall be drawn between the plate and disks at a speed of 10 to 35 ft. per min. The number of holes burned through the specimen shall be counted, each burned hole being considered a conducting path.

### Calculation and Report

47. (a) *Calculation*.—The number of conducting paths shall be calculated as follows:

$$\text{Number of conducting paths per square foot} = \frac{H}{A}$$

where:

$H$  = total number of holes burned in all test specimens, and

$A$  = total number of square feet contacted with the disks.

(b) *Report*.—The report shall include the following:

(1) Number of conducting paths per square foot, and

(2) Number of square feet of paper tested.

### DETERIORATION

#### Apparatus

48. (a) *Baking Oven*.—An electrically-heated oven, equipped with an efficient means of circulating air *within* or *through* the heated chamber, and a suitable fixture upon which the paper specimens may be mounted, shall be used. In case the air is circulated *within* the body of the oven, for example by fan, it is required that the fixture upon which the specimens are mounted

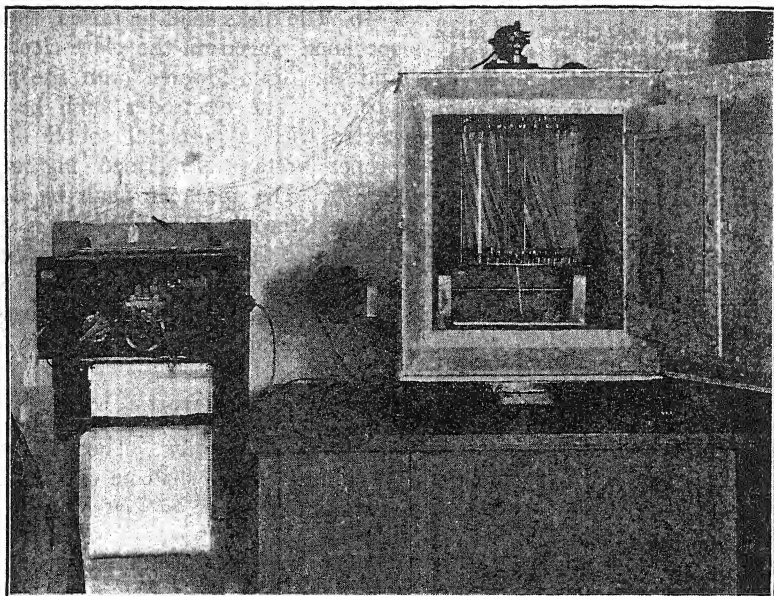


FIG. 6.—Baking Oven Equipped for Circulating Air Within the Oven.

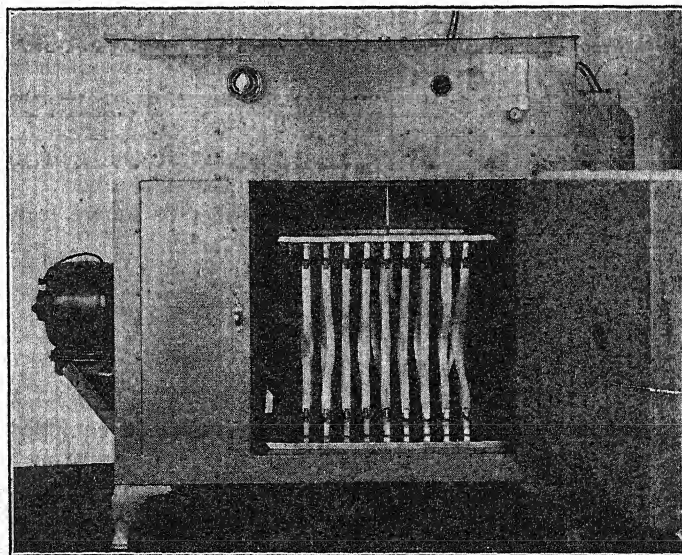


FIG. 7.—Baking Oven Equipped for Circulating Air Through the Oven.

be a turntable rotating within the oven at a speed of about 20 rpm., so that the specimens shall be uniformly exposed to the oven conditions. In the case of heated air circulating *through* the oven, the fixture holding the specimens may be stationary but it is essential that it shall be mounted so as to be at least 4 in. from the oven walls at any point to permit adequate circulation in all parts of the oven, and the design should be such that there shall be an air velocity of at least 1 ft. per sec. through the oven.

(b) *Testing Equipment.*—In addition to the oven it is necessary to provide testing machines for making physical measurements on the baked specimens. The test may be tensile strength, tearing strength, or folding endurance.

### Test Specimens

49. The specimens shall be cut from samples obtained in accordance with Section 2 (a), and shall be at least  $\frac{1}{2}$  in. in width and at least 12 in. in length. At least five groups of five specimens each shall be selected for any of the physical tests noted in Section 48 (b). These groups of specimens shall be selected from the gross sample in some rational manner, so as to represent any known or suspected differences in the quality of the product.

### Procedure

50. (a) The specimens shall be mounted on the fixture as shown in Figs. 6 and 7. In the case of the rotating turntable, Fig. 6, the specimens shall be mounted at such an angle that they will assist, fan-like, in stirring the air within the oven. In the case of a stationary scheme of mounting, Fig. 7, the specimens shall be mounted vertically. The rows of clips supporting the specimens, top and bottom, should be set alternately at 45-deg. angles across the oven so that the air flowing horizontally through the oven would pass between

these staggered rows, turbine fashion, and assist in keeping the temperature uniform in all parts of the oven.

(b) The fixture, readily removable from the oven for mounting the specimens, shall be put in place, and the oven, previously brought to 135 C., shall be closed. Air shall be circulated, as previously indicated, and the temperature shall be maintained at  $135 \pm 2$  C. for 96 hr.<sup>5</sup> It is imperative that the differences in temperature in different parts of the oven shall not exceed 2 C.

### Conditioning and Testing

51. The baked specimens shall be removed from the mounting fixture, care being taken not to damage the strips, and shall be conditioned in a humidity room at a relative humidity of 60 to 65 per cent and at a temperature of 68 to 86 F., for a period of 4 hr. They shall then be tested in this conditioned room for tensile breaking strength, tearing strength, or folding endurance.

### Report

52. The maximum, minimum, and average values shall be reported for each type of test used.

## RATE OF IMPREGNATION

### Scope

53. These methods are intended to apply to papers which are to be impregnated with phenolic varnishes and used in the manufacture of phenol fiber or for various insulating purposes.

### Apparatus

54. The apparatus shall consist of the following:

(a) *Penetrometer.*—A Williams standard tilting type penetrometer with orifice 6.03 cm. (2.375 in.) in diameter as shown

<sup>5</sup> These temperature and time conditions are satisfactory for the more stable types of cable insulating papers. Less severe conditions should be used for other insulating papers and these will be included as soon as they are developed by the committee.

in Fig. 8, or equivalent penetrometer, equipped with a stop watch graduated in seconds.

(b) *Thermometer*.—A Fahrenheit thermometer of suitable range graduated in 0.25 F.

(c) *Oil*.—Mineral or castor oil having a viscosity of  $700 \pm 30$  centipoises at 77 F.

NOTE.—For comparative purposes, liquids other than these may be used.

(d) *Rubber Tubing*.

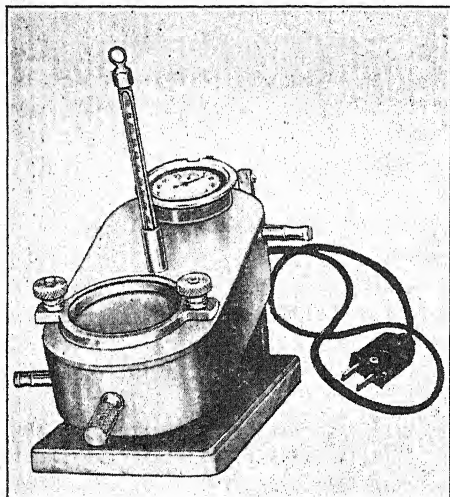


FIG. 8.—Penetrometer.

#### Test Specimens

55. Six test specimens 3 in. square shall be cut from each sample selected in accordance with Section 2 (a). The specimens shall be conditioned as prescribed in Section 3.

#### Procedure

56. (a) The test specimens shall be measured for thickness in accordance with methods A or C of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374)<sup>3</sup>.

(b) The test specimens shall be numbered consecutively on the same side of the paper. The odd-numbered specimens shall be tested with their numbered

sides up, that is, not in contact with the impregnating liquid. The even-numbered specimens shall be tested with their numbered sides down, that is, in contact with the impregnating liquid.

(c) The liquid container shall be placed in a horizontal position and the thermometer shall be so fixed that the bottom of its bulb is approximately  $\frac{1}{4}$  in. above the container bottom. The container shall be filled with the impregnating liquid to within  $\frac{1}{4}$  in. of the upper edge of the container orifice. The liquid shall be maintained at this level by adding small amounts of liquid to replace that absorbed by the test specimens during the testing period. The temperature of the liquid shall be maintained at  $77 \pm 2$  F. during testing by means of the electrical heater or by the circulation of water through the jacket.

(d) The specimen shall be placed over the container orifice and clamped in place with the ring clamp. After setting the stop watch at zero, the container handle shall be depressed quickly until the front end of the container touches the base plate. The surface of the paper shall be observed closely and at the instant of complete penetration of the liquid throughout the entire paper area under test the watch shall be stopped by immediately raising the front end of the liquid container.

(e) The elapsed time shall be recorded as that of impregnation.

#### Report

57. The report shall include the following:

(a) The average, maximum, and minimum times for total impregnation, in seconds,

(b) The average, maximum, and minimum thicknesses before impregnation, in inches, and

(c) Thickness of the specimen showing the maximum impregnation time and also the thickness of the specimen showing the minimum time.



TEST FOR pH OF INSULATING PAPER<sup>6</sup>

## Scope

58. This method of test is designed to indicate the active acidity and alkalinity of aqueous extracts of electrical insulating papers. Such extracts are normally unbuffered and are readily affected by atmospheric carbon dioxide. This procedure embodies features to prevent errors from this cause. The method consists of a hot water extraction of the specimen followed by a pH measurement of the cooled extract solution in an atmosphere of nitrogen. The pH measurement involves the use of a glass-calomel electrode system with suitable potentiometric equipment.

## Apparatus

59. The apparatus shall consist of the following:

- (a) *Bath*.—A hot water bath.
- (b) *Motor and Stirrer*.—A motor with a stirrer constructed as shown in Fig. 2. The stirrer shall be made of brass, chromium plated to render it acid and alkali resistant.
- (c) *Flasks*.—Wide-mouth acid-resistant and alkali-resistant glass<sup>4</sup> 250-ml. Erlenmeyer flasks and wide-mouth flasks of suitable size for use in the electrode assembly described in Paragraph (k); 250-ml. evaporating flasks are satisfactory.
- (d) *Thermometers*.—Thermometers having a range of from 0 to 100 C. and graduated in 1 C. intervals.
- (e) *Electric Hot Plate*.
- (f) *Suction Filtering Apparatus*.
- (g) *Reflux Condenser*.—Water-cooled acid-resistant and alkali-resistant glass.<sup>4</sup>
- (h) *Ascarite or Soda Lime Guard Tube*.
- (i) *Potentiometer Equipment*.—A potentiometer, galvanometer and auxiliary electrical equipment capable of operating

with a limit of error of plus or minus 0.006 v. A direct reading pH meter with manually adjusted temperature compensator is desirable. The meter shall operate with a limit of error of plus or minus 0.1 pH.<sup>7</sup>

(j) *Electrodes*.—A saturated calomel reference electrode with a "salt bridge," and a glass electrode.<sup>8</sup> Commercial glass electrodes containing permanently sealed internal electrodes require no preparation. Open-tube glass electrodes may be prepared by filling the electrode tube with a solution of 0.1 N hydrochloric acid saturated with quinhydrone. A bright platinum wire electrode shall then be inserted in the open tube. The glass electrode and its lead wire shall be suitably shielded to prevent electrostatic interference.

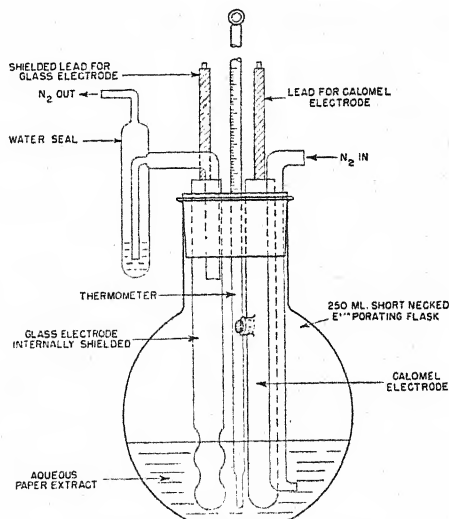


FIG. 9.—Electrode Assembly

(k) *Electrode Assembly*.—The electrode assembly shall consist of a suitable flask provided with a rubber stopper for holding the calomel reference electrode,

<sup>7</sup>This potentiometer equipment is available from several manufacturers.

<sup>8</sup>This equipment is available from several manufacturers. For the electrode assembly shown in Fig. 9, electrodes approximately 5 in. in length are satisfactory.

<sup>6</sup>This test was developed by the Joint D-6—D-9 Committee on pH Problems.

the glass electrode, the thermometer, a gas inlet tube extending to the bottom of the flask and a gas outlet tube. An arrangement that allows removal of the flask without disturbing the electrodes, thermometer, and gas inlet and outlet tubes is desirable. A satisfactory assembly is shown in Fig. 9.

NOTE.—As a precaution against possible carbon dioxide contamination from the atmosphere, unnecessary transference of the extract solution from one flask to another should be avoided where practicable. It is, therefore, desirable that the flask selected for use in the electrode assembly shall also be suitable for the reflux and cooling operations specified in Section 63. Flasks and condensers fitted with ground-glass joints are convenient and preferable when available.

### Reagents

60. (a) *Standard Buffer Solution (for standardizing the glass electrode).*—Dry about 6 g. of analytical grade potassium acid phthalate for not less than 2 hr. at 120 C. Cool in a desiccator containing a good desiccant. Add 5.0905 g. of the salt to 500 ml. of distilled water at 25 C. The pH of this buffer solution is taken as 4.0 at 25 C.

(b) *Distilled Water.*—The boiled distilled water shall have a pH of 7.0 to 8.0 when boiled and tested in accordance with the procedure prescribed in Section 63 in the absence of a paper sample. Water meeting this requirement may be prepared by double distillation, the second distillation being over alkaline permanganate. Acid-resistant and alkali-resistant glass apparatus should be used for these distillations.

### Characteristics of Electrode System

61. (a) *Preparation for Use and Maintenance.*—The electrodes require regular attention to obtain constant and reproducible values. Before and after using, wipe them with a piece of lens paper or soft cloth. The calomel elec-

trode shall contain at all times sufficient saturated potassium chloride so that the level of the salt solution inside the electrode is always above the level of the liquids in which it is immersed. When not in use, keep the lower half of each electrode immersed in distilled water. Before placing the electrodes in the solutions to be measured, rinse them several times with fresh portions of distilled water until a constant pH is obtained. This precaution is especially important after the buffer standardization.

(b) *Calibration.*—To calibrate the electrodes, place sufficient standard buffer solution at  $25 \pm 1$  C. in a beaker to cover the lower half of the electrodes. If a direct reading pH meter is employed adjust the temperature compensator for the temperature of the solution and vary the "asymmetry potential" adjustment until the meter registers a scale reading of 4.0 while the electrodes are immersed in the buffer solution. With other forms of potentiometric equipment, read and record the potential. In operations over extended periods of time, the electrode calibration should be verified frequently.

### Test Specimen

62. From the samples obtained in accordance with Section 2(a), a composite test specimen weighing at least 5 g. shall be cut into small pieces approximately 1 cm. (0.4 in.) square. The specimen shall be thoroughly mixed and, during preparation, any contamination by handling shall be avoided.

### Procedure

63. (a) A 1-g. portion of the composite specimen shall be placed in the 250-ml. Erlenmeyer flask and 100 ml. of boiling distilled water added. The flask shall then be clamped in position in a boiling water bath so that at least one-half of

the flask is immersed in the water bath. During the stirring, the temperature of the contents of the flask shall remain at least as high as 95 C. The stirrer shall be mounted so that the blades are within 1 cm. (0.4 in.) of the bottom of the flask. The assembled extraction apparatus is shown in Fig. 3. The stirrer shall be driven at a speed of 4000 to 5000 rpm. for 5 min. At the end of this period the specimen should have been thoroughly pulped. For paper unusually difficult to pulp, the period of stirring shall be increased to 10 min.

(b) Immediately after the specimen has been pulped, the contents of the flask shall be filtered rapidly with suction through a perforated porcelain disk, refiltering the first portion of the filtrate to permit the formation of a mat. The residual pulp shall not be washed.

NOTE.—It is important that the filtration of the extract be accomplished as promptly as possible after the disintegration. When the fibers are too short to form a satisfactory mat on the perforated porcelain disk, filter with suction through a fine quantitative filter paper (which has been washed twice with 100-ml. portions of hot distilled water) on a Buchner funnel.

(c) After the filtration, the extract solution shall be diluted to 100 ml. with hot distilled water by bringing it up to the mark in a 100-ml. graduated cylinder. It shall then be transferred to the electrode assembly flask.

(d) The flask containing the filtered extract shall be attached to the reflux condenser by means of a rubber stopper covered with tin or aluminum foil, and boiled for 5 min. to remove traces of absorbed carbon dioxide.

(e) At the end of the boiling period, the ascarite or soda lime guard tube shall be attached to the exposed end of the condenser. After being protected in this way against atmospheric carbon dioxide, the extract solution shall be al-

lowed to cool to room temperature. The cooling time may be shortened by immersing the flask in a cooling bath.

NOTE.—When a series of tests are being made, the reflux and cooling operations may be facilitated by attaching an air-cooled condenser to the flask prior to the cooling period. This will make the water-cooled condenser available for the refluxing of additional extract solutions. A 10-mm. glass tube approximately 450 mm. in length is satisfactory for this purpose; it may be fitted to the flask by means of a foil-covered rubber or cork stopper. The exposed end of the condenser should be fitted with an ascarite or soda lime guard tube.

(f) After the extract solution has cooled, the condenser shall be removed from the flask and the flask shall be attached immediately to the electrode assembly. During this operation nitrogen gas shall be passed through the bubbling tube. The nitrogen bubbling shall be continued for 10 min.

NOTE.—Because the effect of carbon dioxide becomes negligible in acid solution, Paragraphs (d), (e) and the nitrogen bubbling of Paragraph (f) may be omitted when an extract is known to have a pH lower than 5.3 as measured by this method.

(g) After the 10-min. period and with the nitrogen still bubbling through the solution, the temperature of the solution shall be adjusted to  $25 \pm 1$  C. When a direct reading pH meter is being used, the temperature compensator shall be adjusted for the temperature of the solution and the pH shall be read. When any other form of potentiometric equipment is employed, the voltage shall be read.

(h) At least two specimens shall be tested. If the pH readings on duplicate determinations do not agree within 0.3 pH unit, the determinations shall be repeated.

#### Calculation and Report

64. (a) *Calculation.*—With potentiometric equipment which does not record the pH directly, the pH of the extract



solution shall be calculated as follows:  $t$  = temperature in degrees Centigrade of extract solution at time of measurement, and

$$\text{pH} = \frac{(E_2 - E_1) \times 5039.4}{273 + t} + \text{pH}_1$$

where:

$E_1$  = voltage recorded with standard buffer solution,

$E_2$  = voltage recorded with extract solution,

$\text{pH}_1$  = nominal pH of standard buffer solution.

(b) *Report.*—The results shall be reported to the nearest 0.1 pH unit.

# *Tentative Specifications for*

## NATURAL BLOCK MICA AND MICA FILMS SUITABLE FOR USE IN FIXED MICA-DIELECTRIC CAPACITORS<sup>1</sup>



**A.S.T.M. Designation: D 748 - 45 T**

ISSUED, 1943; REVISED, 1945.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These specifications cover natural block mica and mica films suitable for use in the manufacture of fixed mica-dielectric capacitors.

**NOTE.**—The properties included in these specifications are those required to identify the types of natural block mica and mica films suitable for use in the manufacture of fixed mica-dielectric capacitors. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available. Natural block mica and mica films which do not conform to the requirements of these specifications for capacitor use may well be capable of meeting the requirements for other critical electrical insulating purposes, such as in vacuum tubes, and airplane spark plugs.

(b) The system of classifying quality of natural block mica and mica films, covered by these specifications, and which has been tried out commercially

and found to be both practicable and reliable, is based on a combination of electrical and physical properties which the mica must possess. This system differs radically from past practices and previous concepts of mica quality for capacitor use. It does not discriminate against the presence of spots and stains in even first quality electrically selected mica providing the mica conforms to specific electrical and physical requirements. Appreciable amounts of air inclusions and waviness also are permitted in all electrical quality classes providing the mica meets specific electrical and physical requirements. However, such mica is not considered generally as desirable as mica having lesser amounts of such defects. Mica capable of meeting specific requirements also may be of any basic color such as white, ruby, light green, dark green, brownish green, rum, or other colors derived from any source. As the electrical system of classification qualifies mica previously considered visually unsuitable for capacitors, the mica supply for such use is greatly increased as compared with the

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, June 27, 1945.

choicer visual quality types. A somewhat lower yield (10 to 20 per cent) of films from electrically selected block mica may be experienced with lower visual quality micas due principally to poorer splitting properties or improper preparation of the block. Appendix III gives approximate "visual quality" equivalents determined in accordance with the Standard Methods of Testing, Grading, and Classifying Natural Mica (A.S.T.M. Designation: D 351).<sup>3</sup>

TABLE I.—GRADES (SIZES) OF NATURAL BLOCK MICA OR MICA FILM.

Grade	Area of Rectangle, sq. in.	Minimum Dimension of One Side, in.
OEE Special.....	100 and over	..
OEE Special.....	80 to 100	..
EE Special.....	60 to 80	..
Extra Special.....	48 to 60	..
A-1 (Special).....	36 to 48	4
1.....	24 to 36	3
2.....	15 to 24	2
3.....	10 to 15	2
4.....	6 to 10	1½
5.....	3 to 6	1
5½.....	2½ to 3 <sup>a</sup>	1
6.....	1 to 2½	¾

<sup>a</sup> 1943 India Mica Mission permits 2½ to 3 sq. in.

## Forms

2. These specifications cover the following two forms of natural mica, suitable for use in the manufacture of mica-dielectric capacitors:

*Form 1.*—Full-knife trimmed natural block mica 0.007 to 0.030 in. in thickness.

NOTE.—A certain percentage of block mica thinner than 0.007 in. may be accepted under these specifications, subject to agreement between the purchaser and the manufacturer.

*Form 2.*—Films or capacitor splits 0.0008 to approximately 0.004 in. in thickness from trimmed block mica or cut to size.

## Grades (Sizes)

3. The grade or size of block mica or films split from trimmed block mica shall be based on the usable area of the rectangle which can be cut from the specimen and the minimum dimension of one side. The mica in the usable rectangle shall conform to the quality requirements specified by the purchaser. The grades or sizes of block mica or mica films covered by these specifications shall be based on the so-called India grading scale, in general use for many years, as given in Table I.

## Classes

4. These specifications cover the following three classes of natural block mica or mica films. The class of mica desired shall be specified by the purchaser. Block mica or mica films shall conform to all of the requirements of its respective class, unless otherwise specified by the purchaser.

*Class C-1.*—Class C-1 block mica or mica films has the highest *Q* value obtainable for mica (2500 minimum in capacitors) and is suitable for use in all sizes and types of silver and foil electrode molded and clamped unit capacitors, including the most critical types, for use in high stability tuned circuits, as well as high current radio frequency capacitors used in radio transmitter circuits (Note).

NOTE 1.—Based on commercial experience (see Appendix II), class C-1 block mica or mica films is satisfactory for the manufacture of all of these types of mica capacitors. However, it has been found that "medium to heavy" air-stained mica may produce a slightly lower yield of highest stability and high current radio frequency types of capacitors as well as a somewhat lower yield of satisfactory silver electrode mica laminations. Likewise, "medium to heavy" wavy mica may also adversely influence the application of silver electrodes. In addition, there is some possibility that excessive waviness may cause cracked laminations in molded capacitors due to the high molding pressures employed and it may be less suitable from a stacking

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE II.—ELECTRICAL, PHYSICAL, AND VISUAL QUALITY REQUIREMENTS OF NATURAL BLOCK MICA AND MICA FILMS FOR USE IN CAPACITORS.

	Class C-1	Class C-2	Class C-3
<b>Electrical Properties:</b>			
Conductivity.....	none	none	none
Q value or power factor at 1 megacycle.....	E-1 <sup>a</sup>	E-2 <sup>a</sup>	E-3 <sup>a</sup>
Dielectric strength, v. per mill at 60 cycles per sec., min.,	average...	1000	1000
	single.....	850	850
	b	b	b
Dielectric constant.....			
<b>Physical Properties:</b>			
Weight loss on heating (5 min. at 600 C.), max., per cent.....	0.2	0.2	...
Thickness uniformity (mica films) <sup>c</sup> .....	best	best	intermediate
Temperature coefficient of capacitance and retrace.	d	d	d
<b>Visual Qualities:</b>			
Air inclusions <sup>e</sup>	A.....	none to slight	none to slight
	B.....	medium	medium
	C.....	medium to heavy	medium to heavy
Waves <sup>f</sup>	A.....	flat to slight	flat to slight
	B.....	medium	medium
	C.....	medium to heavy	medium to heavy
Cracks.....	none	none	none
Tears.....	none	none	none
Pin holes.....	none	none	none
Stones.....	none	none	none
Buckles.....	none	none	none
Ridges.....	none	none	none

<sup>a</sup> The Q value or power factor ( $Q = \frac{1}{\text{power factor}}$ ) of block mica or mica films suitable for use in capacitors shall fall within the three electrical quality groups, based on end-use requirements, designated E-1, E-2, or E-3. These quality groups shall conform to the Q or power factor values prescribed in Table III or the corresponding scale readings of the vacuum-tube voltmeter when tested by the rapid, direct-reading method described in Appendix I.

<sup>b</sup> As the dielectric constant of natural block mica suitable for use in capacitors is fairly uniform, no specified requirement is needed.

<sup>c</sup> Until definite values can be specified, the permissible amounts of such defects shall be agreed upon by the purchaser and the manufacturer.

<sup>d</sup> It has been found that the temperature coefficient of capacitance and retrace of capacitors made with Classes C-1, C-2, and C-3 mica are more dependent on such factors as electrical and mechanical design and manufacturing technique than any differences that may be attributed to the mica itself.

<sup>e</sup> The amount of air inclusions shall not exceed the specified limits for each subclass as judged by the photographic reference standards<sup>4</sup> shown in Figs. 1 to 3. The permissible amount of air inclusions shall be stated by suffixing the letter A, B, or C, as the case may be, to the required electrical quality class.

<sup>f</sup> Until definite values can be specified, the permissible amount of waves, buckles and ridges shall be agreed upon by the purchaser and the manufacturer. The permissible amount of waves shall be stated by suffixing the letter A, B, or C, as the case may be, to the letter denoting the amount of permissible air inclusions, for example: Class C-1 B A block mica or mica films denotes:

C-1	B	A
Best electrical quality	medium air inclusions	flat to slight waves

standpoint. Similarly, some reduction in unit volume capacitance of foil electrode capacitors may result from excessive amounts of either air inclusions or waviness.

**Class C-2.**—Class C-2 block mica and mica films has a high order of Q value (1500 minimum in capacitors) and is suitable for use in all sizes and types of silver and foil electrode molded and clamped unit capacitors similar to those specified for class C-1 mica. However, a certain percentage of capacitors made with class C-2 block mica and films may show a somewhat higher temperature rise in transmitter types than capacitors made with class C-1 block mica or mica films.

**Class C-3.**—Class C-3 block mica and mica films has the lowest Q value (minimum 200 in capacitors) of the three classes covered by these specifications. Such Q value, however, is sufficiently high to permit this mica to be classed as a low-loss insulating material. This mica is particularly suitable for use in foil electrode molded and clamped type capacitors (Note) used in less critical circuits for blocking and coupling purposes where high Q value, high stability and low temperature coefficient are not required.

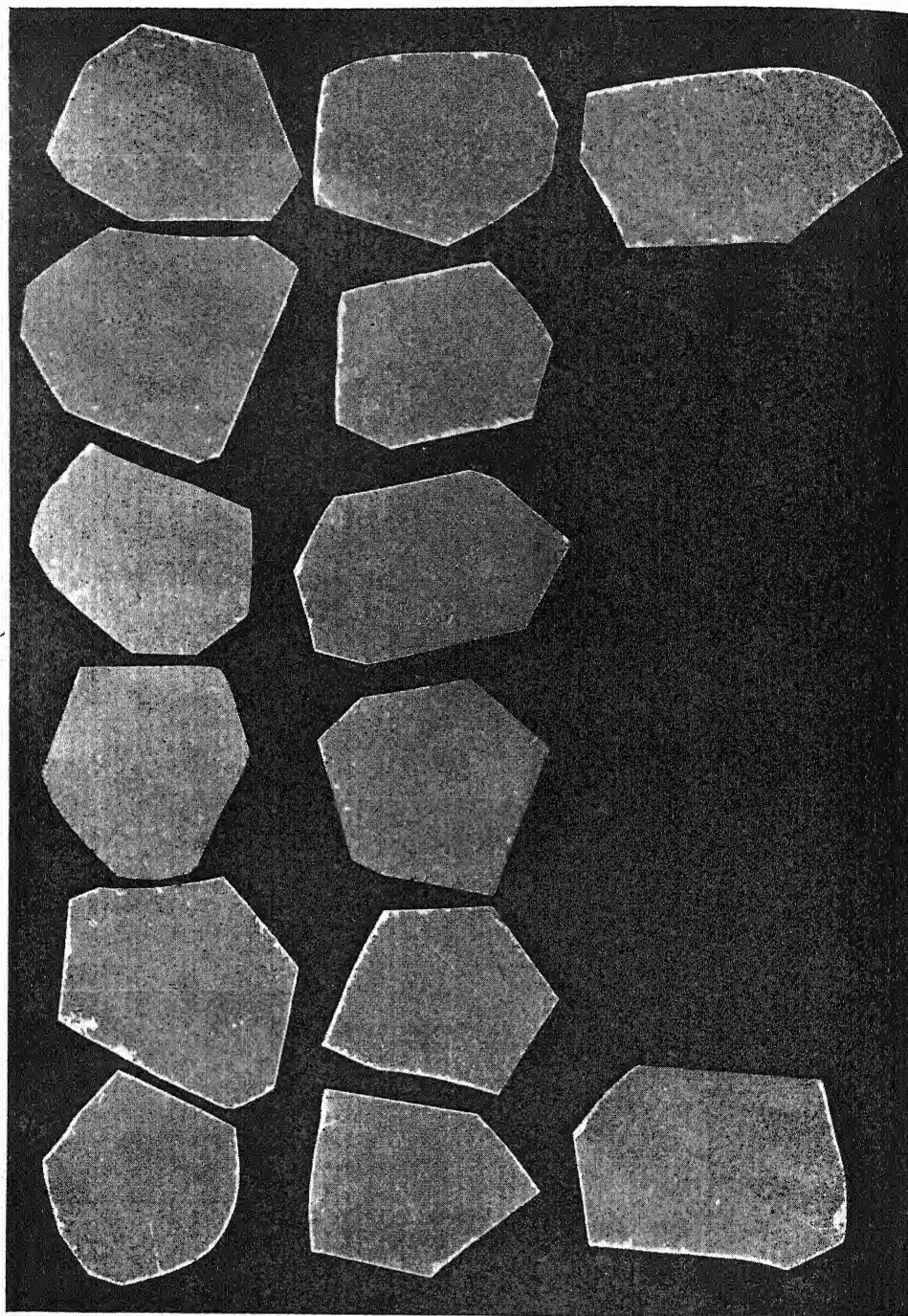
NOTE 2.—Experience has shown that silver electrode molded capacitors made with class C-3 mica which contained slightly conducting spots and stains but contained “none to slight” air inclusions and “flat to slight” waves, had temperature coefficient and capacitance stability characteristics just as good as obtained with capacitors made with the best class C-1 mica.

### Electrical and Physical Properties, and Visual Qualities

5. Natural block mica and mica films shall conform to the requirements as to electrical and physical properties and visual qualities as prescribed in Table II.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in



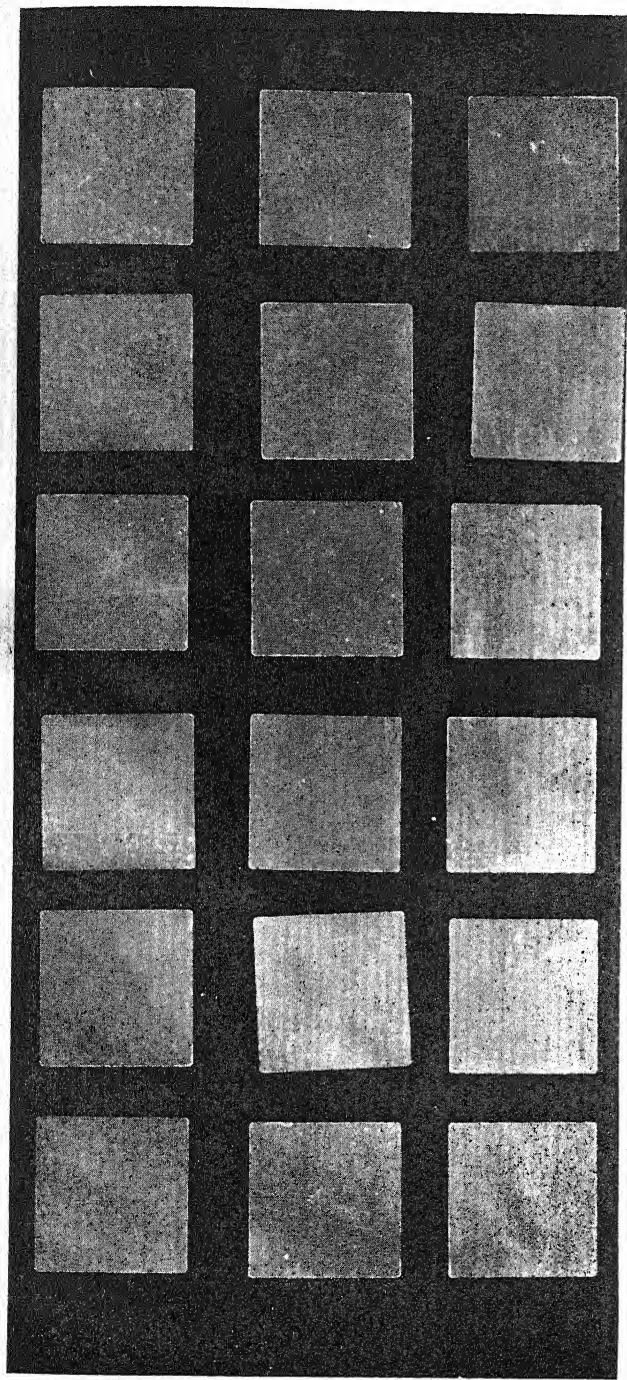
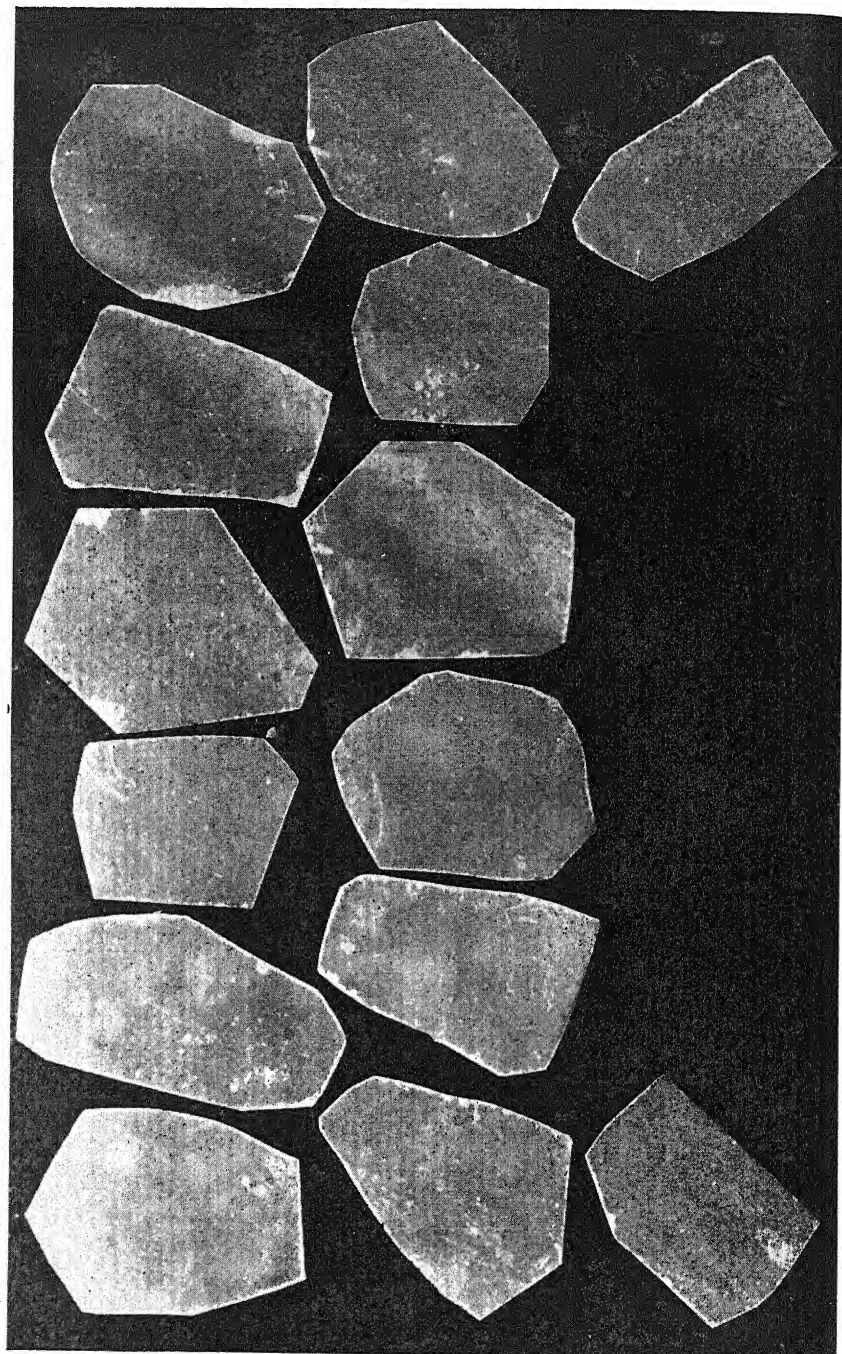


FIG. 1.—Reduced Facsimile of Mica Films and Cut Mica Films (0.0012 to 0.002 in. in Thickness) Showing Maximum Allowance Air Inclusions for Quality A.<sup>4</sup>

<sup>4</sup> Glossy prints of the original photographic reference standards are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.





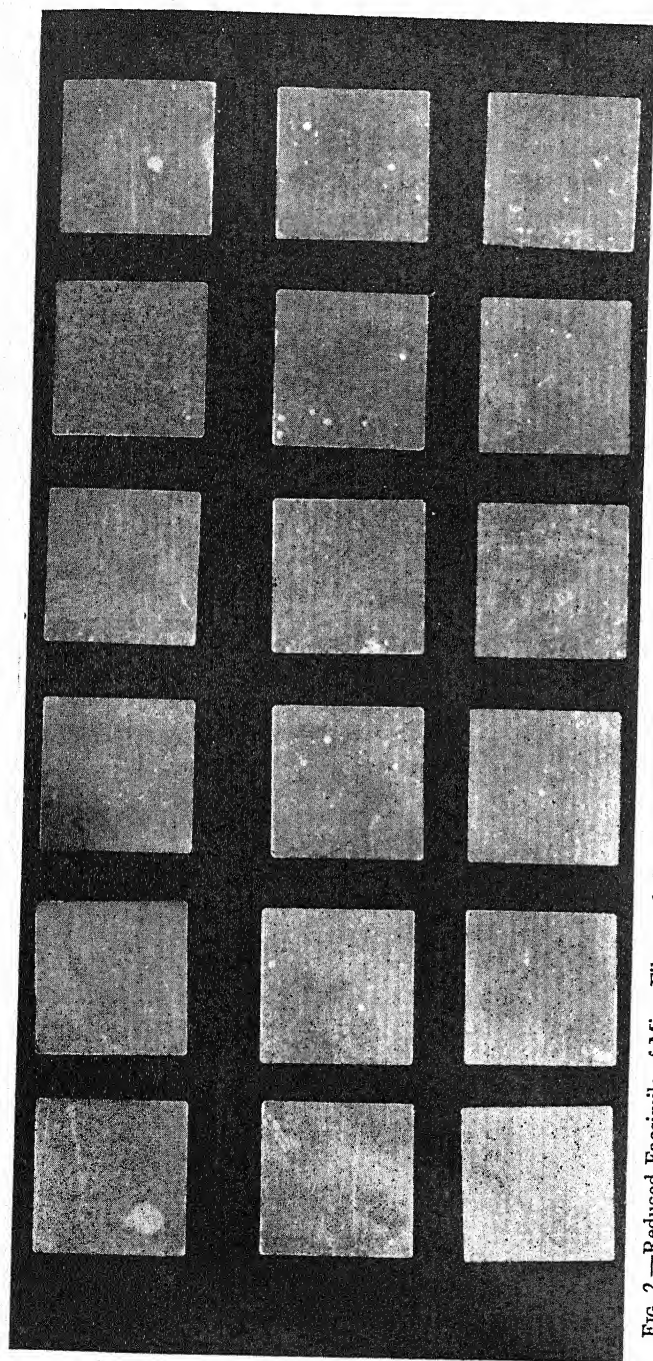
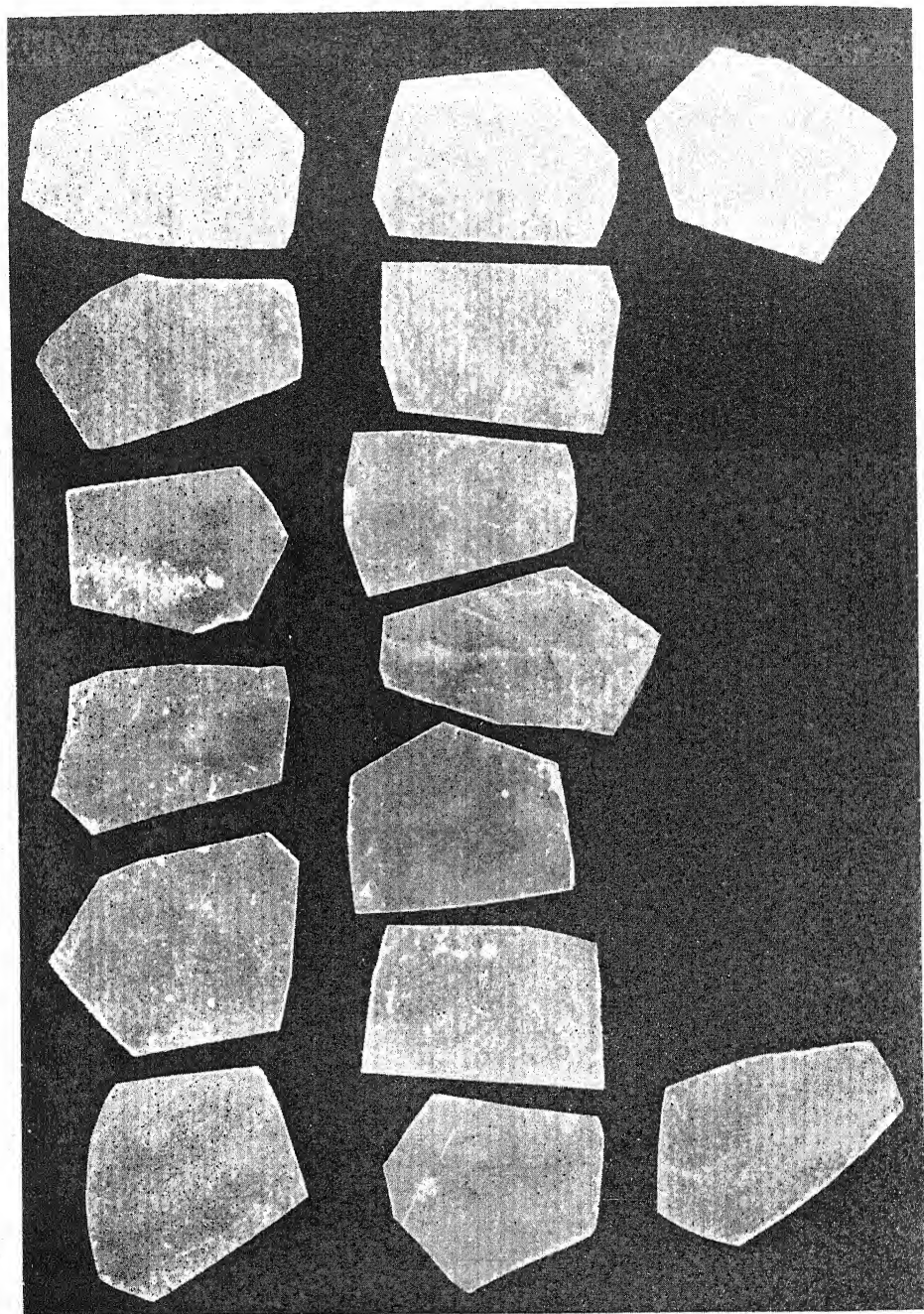


FIG. 2.—Reduced Facsimile of Mica Films and Cut Mica Films (0.0012 to 0.002 in. in Thickness) Showing Maximum Allowable Air Inclusions for Quality B.<sup>4</sup>





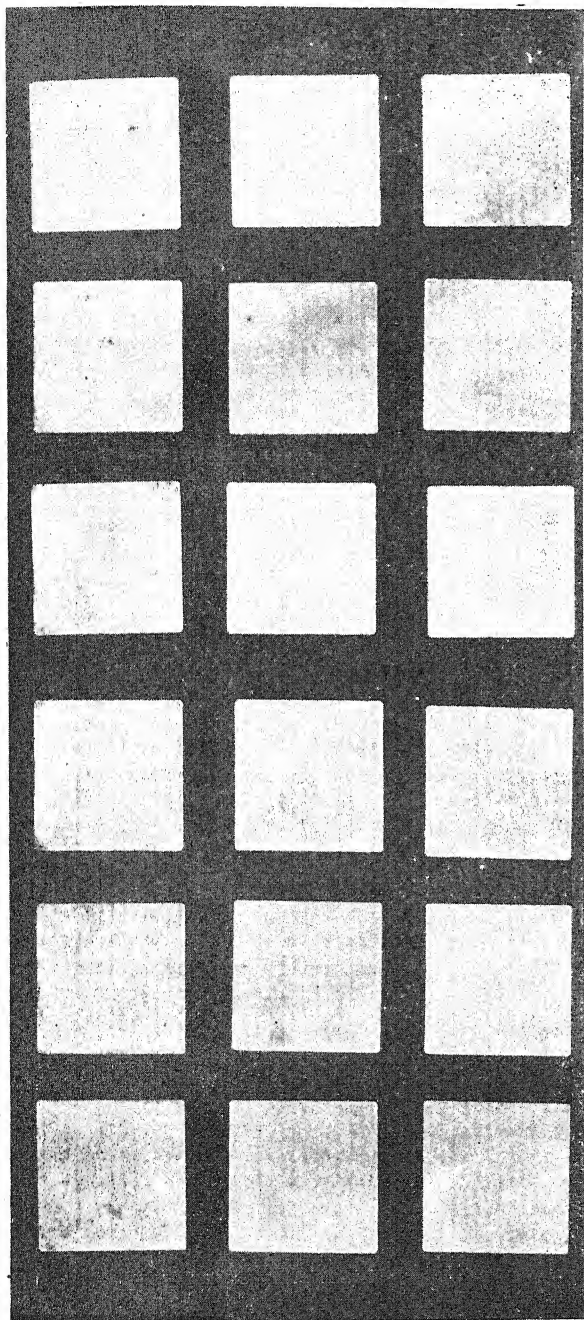


FIG. 3.—Reduced Facsimile of Mica Films and Cut Mica Films (0.0012 to 0.002 in. in Thickness) Showing Maximum Allowable Air Inclusions for Quality C.<sup>4</sup>

accordance with the following methods of test:

(a) *Grading According to Size.*—Standard Methods of Testing, Grading, and Classifying Natural Mica (A.S.T.M. Designation: D 351),<sup>3</sup> Section 2 and A.S.T.M. Chart in Plate I of Methods D 351.

(b) *Electrical Conductivity.*—Standard Methods of Test for Conducting Paths in Electrical Slate (A.S.T.M. Designation: D 273),<sup>3</sup> using a spark coil test set except that a sharp-pointed electrode shall be used in place of the comb electrode for exploring for conducting veins

films. In this instance the purpose of the test is to detect dielectric weakness due to any cause.

(c) *Q Value or Power Factor.*—A.S. T.M. Methods D 351, at 1 megacycle, or by the rapid, direct-reading method described in Appendix I of these specifications.

NOTE 2.—Information available indicates that block mica found suitable for use at 1 megacycle, is equally or more suitable for use at higher frequencies. However, such mica may or may not be as suitable for use at lower frequencies. In critical low-frequency applications it therefore may be desirable to test at a frequency lower than 1 megacycle.

TABLE III.—Q AND POWER FACTOR VALUES FOR ELECTRICAL QUALITY GROUPS E-1, E-2, AND E-3.

Q or Power Factor Group	Form	Q Value	Power Factor	Rapid Method Meter Reading		
				0.010 in. <sup>c</sup> (0.007 to 0.015 in.)	0.020 in. <sup>c</sup> (0.015 to 0.025 in.)	0.030 in. <sup>c</sup> (0.025 to 0.035 in.)
E-1.....	Block mica..... Mica films <sup>b</sup> .....	2500 min. 2500 min. <sup>a</sup>	0.0004 max. 0.0004 max.	95 to 100 95 to 100	95 to 100 95 to 100	95 to 100 95 to 100
E-2.....	Block mica..... Mica films <sup>b</sup> .....	350 to 2500 1500 min. <sup>a</sup>	0.00285 to 0.0004 0.00066 max.	87 to 95 ...	77 to 95 ...	71 to 95 ...
E-3.....	Block mica..... Mica films <sup>b</sup> .....	50 to 350 200 to 1500 <sup>a</sup>	0.02 to 0.00285 0.005 to 0.00066	50 to 87 ...	32 to 77 ...	24 to 71 ...

<sup>a</sup> Extensive commercial tests have verified the validity of the Q values of capacitors made with group E-1, E-2, and E-3 block mica to a satisfactory degree.

<sup>b</sup> Minimum Q values for material purchased as films and tested in stacks of films 0.010 to 0.030 in. in thickness. These also indicate probable minimum Q values of molded type, 1000 mmf., stacked-foil, and silvered capacitors. (See Fig. 6 in Appendix I.) These will apply when all factors that would adversely influence the Q value are under control.

<sup>c</sup> Thickness of block mica or mica films stacked to this thickness.

and spots. This test shall be made in subdued light in a lighted room.

NOTE 1.—For the purpose of these specifications, electrical conductivity in spotted and stained areas of block mica is revealed when visible sparking or glowing takes place inside or on the surface of the mica in the vicinity of the test probe and not by actual puncture of the mica by the high-potential current. If actual puncture of the test specimen does take place this indicates the presence of mechanical faults, such as, pin holes, tears, or cracks which extend completely through the mica. While this test method has been found suitable for controlling conductivity in spots and stains and dielectric weakness due to mechanical faults in block mica, an even greater factor of safety will be realized if this flash test is applied directly to capacitor

(d) *Dielectric Strength.*—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S. T.M. Designation: D 149),<sup>3</sup> using the short-time test with  $\frac{1}{4}$ -in. diameter electrodes in oil.

NOTE 3.—In cases of dispute arising from borderline cases of Q value or power factor and dielectric strength, the test specimens shall be baked for a minimum period of 2 hr. at a temperature of 121 C. (250 F.), and tested immediately upon cooling to room temperature.

(e) *Weight Loss on Heating.*—Test specimens shall be preheated in an oven

at 121 C. (250 F.) for a minimum time of 2 hr. and then weighed. The specimens shall then be heated in the oven at 600 C. (1110 F.) for 5 min. and reweighed. The percentage loss in weight after heating shall be calculated based on the weight of the specimen at the end of the 2-hr. preheating period.

(f) *Thickness Uniformity (Films).*—Splitting quality shall be judged by the uniformity of thickness of films split from block mica by viewing between crossed polaroids.

(g) *Visual Qualities.*—The visual qualities of block mica shall be judged under the following conditions:

*Air Inclusions.*—Reflected daylight or equivalent.

*Waves, Buckles, and Ridges.*—Reflected daylight through a window frame where distortion of parallel and vertical lines of reflected image can be judged.

NOTE.—Quantitative methods of test for these qualities are under development.

*Cracks, Tears, Pinholes, and Stones.*—The presence of such mechanical defects as cracks, tears, pinholes, and stones shall be determined by the spark coil test as prescribed in A.S. T.M. Methods D 273.

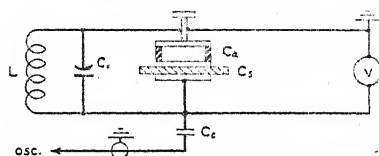
## APPENDIX I

### RAPID, DIRECT-READING, RESONANT-CIRCUIT METHOD FOR DETERMINING $Q$ VALUE OR POWER FACTOR OF NATURAL BLOCK MICA OR MICA FILMS

A1. *General.*—This Appendix contains a description of a rapid, direct-reading, resonant-circuit method for measuring power factor or  $Q$  value of sheet insulating materials at a frequency of 1 megacycle, although it has been found equally suitable for such purpose over a frequency range from 50 kc. up to 100 megacycles. It is not intended as a precision method. Its chief virtues are the speed and simplicity with which a useful power factor or  $Q$  value indication can be obtained. No special preparation of the test specimen is necessary (other than conditioning, where necessary), such as the application of tin-foil electrodes. Likewise, no calculations are necessary.

A2. *Theory of Operation.*—This method makes use of a comparison between the maximum resonant voltage measured across a low-loss capacitor in parallel with an inductance and the maximum resonant voltage across the same circuit when a piece of block mica is introduced in series with the low-loss capacitor. To accomplish this, resonance is reestablished each time a test specimen is placed in series with the low-loss capacitor by adding capacitance to duplicate the initial capacitance of the resonant circuit, by means of a variable vernier air capacitor, in parallel with the coil. This provides a test method requiring but a single dial adjustment and this combined with a direct-reading meter,

greatly facilitates rapid testing. Greater testing speed can be realized by adopting this method to full automatic means.



- $L$ —Inductance having a  $Q$  value of at least 300 at 1 megacycle.
- $C_v$ —Vernier capacitor (approximately 10 mmf. maximum).
- $C_a$ —Fixed low-loss air capacitor (approximately 20 mmf.).
- $C_2$ —Test specimen.
- $C_c$ —Input coupling capacitor.
- $V$ —Vacuum tube voltmeter.

FIG. 4.—Diagram of Connections of Test Circuit for Measuring Power Factor.

A3. *Calibration.*—It is possible to calibrate the indicating scale of the vacuum-tube voltmeter directly in terms of power factor or  $Q$  value by using test specimens of block mica of known power factor. The resulting scale then can be used to give a direct indication of power factor or  $Q$  value over a useful range. As the meter indication is a function of both the power

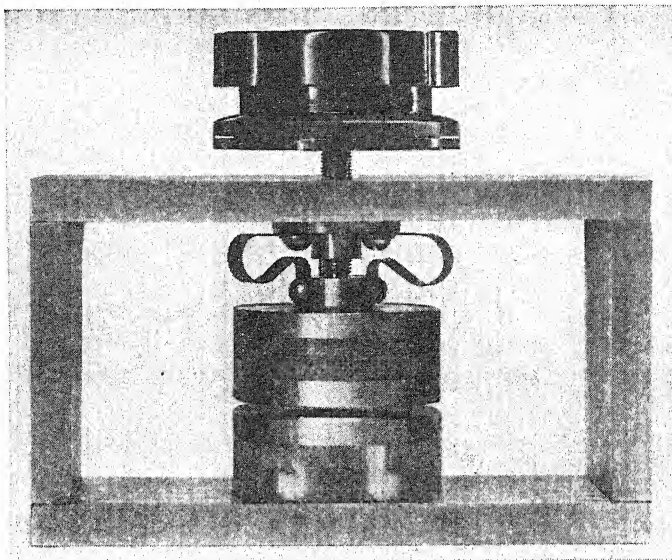


FIG. 5.—Test Electrodes.

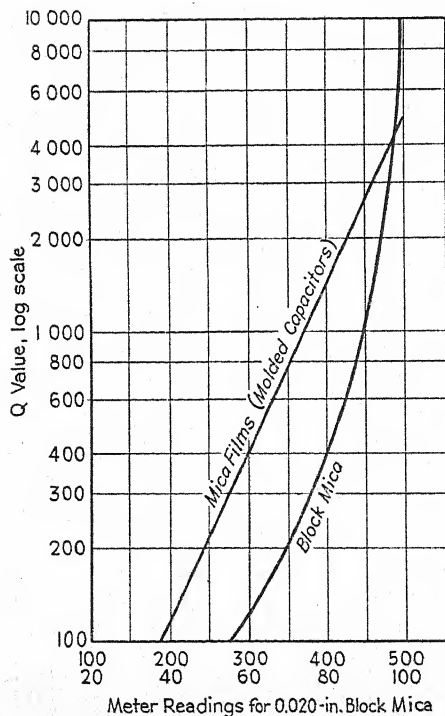


FIG. 6.—Typical Calibration Curves of Block Mica Determined by the Rapid, Direct-Reading Method.

factor and the thickness of the test specimen, the latter also shall be taken into consideration in the calibration. It has been found that if the inductance coil used in such a test circuit has a  $Q$  value of approximately 300, the  $Q$  range will extend from somewhere in the vicinity of 10,000 at full scale to about 50 at quarter scale of the indicating meter. It further has been found that using inductances with  $Q$ 's of 300 or more, the calibration is fairly independent of frequency and a frequency range from 50 kc. to 50 megacycles can be covered by merely changing the inductances and the oscillator test frequency. Even without a true  $Q$  value calibration this method is capable of giving relative values of  $Q$  on an arbitrary scale where such indications are all that might be required. It has been found feasible to use an arbitrary scale in the measurement of the  $Q$  value of block mica.

A4. *Test Circuit*.—A diagram of connections of the test circuit is shown in Fig. 4.

A5. *Sensitivity*.—High sensitivity in the low power factor or high  $Q$  value range is a function of high circuit  $Q$  value. To obtain this, particular care is necessary in the choice of components and insulating materials. For test frequencies of 1 megacycle and lower,  $C_v$  (Fig. 4) may be a high quality conventional type variable air capacitor with a single movable plate. For frequencies above 1 megacycle the vernier capacitor should, preferably, form an integral



part of the test electrodes so as to keep series resistance and inductance effects at a minimum.<sup>5</sup>

A6. *Test Electrodes.*—The test electrodes<sup>6</sup> found satisfactory for this test method are shown in Fig. 5. These test electrodes have been designed with a view of adopting them to standard commercial testing equipment incorporating an oscillator and a vacuum-tube voltmeter.

A7. *Typical Calibration Curves.*—A typical calibration curve for the  $Q$  value of block mica and one in terms of probable  $Q$  value of manufactured capacitors are shown in Fig. 6.

A8. *Meter Scale.*—The calibration of the

meter scale for block mica is based fundamentally on the probable  $Q$  values of capacitors when made from splittings from block mica. Group E-1 corresponds to capacitors having  $Q$  values of 2500, minimum. The range for group E-2 corresponds to capacitors having  $Q$  values of 1500 to 2500, and the E-3 range to capacitors with  $Q$  values up to 1500 at a frequency of 1 megacycle. The minimum  $Q$  value for the E-3 range that capacitors may have appears to be in the neighborhood of 200. The divergence of the  $Q$  value of block mica from capacitor  $Q$  values is an interesting phenomenon. A depreciation from the value for block mica in the high  $Q$  range and an improvement over block mica in the low  $Q$  range when made into molded capacitors has been established. A possible explanation is the contributing losses in the molded casing and the resistance of foil and leads in the first case and the release of imprisoned moisture on splitting and baking in the second case.

<sup>5</sup> See Section 28 (b) of the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150), see p. 667.

<sup>6</sup> Blueprints of detailed drawings for the construction of these test electrodes are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

## APPENDIX II

### RECOMMENDED USE OF BLOCK MICA AND MICA FILMS PER A.S.T.M. SPECIFICATIONS D 748 IN FIXED MICA-DIELECTRIC CAPACITORS PER JOINT ARMY-NAVY SPECIFICATION JAN-C-5

Block Mica and Mica Films per A.S.T.M. Specifications D 748	Capacitors per Joint Army-Navy Specification JAN-C-5
Class C-1 (Within maximum and minimum limits specified for air inclusions and waviness)	All types and sizes of silver and foil electrode molded and potted capacitors of characteristics A to F.
Class C-2 (Within maximum and minimum limits specified for air inclusions and waviness)	Same as above for class C-1, except a small percentage may fail to meet requirements for $Q$ value. Temperature coefficient and retrace and radio frequency temperature rise
Class C-3 (Within maximum and minimum limits specified for air inclusions and waviness)	All sizes of foil electrode characteristic A capacitors.

## APPENDIX III

### APPROXIMATE EQUIVALENTS BETWEEN ELECTRICAL QUALITY PER A.S.T.M. SPECIFICATIONS D 748 AND VISUAL QUALITY PER A.S.T.M. METHODS D 351.<sup>a</sup>

Electrical Quality per A.S.T.M. Specifications D 748	Visual Quality per A.S.T.M. Methods D 351 <sup>b</sup>	
Block Mica and Mica Films	Block Mica	Mica Films
Class C-1 AA.....	Fair stained (min.)	First quality
Class C-1 BB.....	Good stained	Second quality
Class C-1 CC.....	Stained	Third quality

<sup>a</sup> As determined by the Standard Methods for Testing, Grading, and Classifying Natural Mica (A.S.T.M. Designation: D 351).<sup>3</sup>

<sup>b</sup> Mica film quality is not specified in A.S.T.M. Methods D 351. The qualities given are in commercial use and the equivalent block mica quality is assumed.

The visual quality equivalents are valid only when the  $Q$  value of the mica is E-1 when determined in accordance with A.S.T.M. Specifications D 748. Experience has shown that the  $Q$  value range of ruby and white types of block mica, regardless of source, is 80 to 95 per cent E-1, whereas, the  $Q$  value range of light green, dark green, greenish brown, and rum-colored block mica is 45 to 90 per cent E-1. It is important to bear in mind that it is permissible for all qualities of mica prescribed in A.S.T.M. Specifications D 748, to contain spots and stains, providing the mica meets all electrical and physical requirements, whereas, no spots or stains (other than air stains) are permitted in the above visual quality groups per A.S.T.M. Methods D 351.

# TENTATIVE METHODS OF TESTING AND TOLERANCES FOR JUTE ROVE AND PLIED YARN FOR ELECTRICAL AND PACKING PURPOSES<sup>1</sup>



A.S.T.M. Designation: D 681 - 42 T

ISSUED, 1942.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods of testing and tolerances apply to plied jute yarn and to single and plied jute rove, both treated and untreated, for electrical and packing purposes.

## Definitions

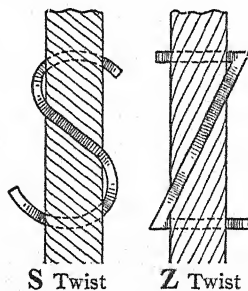
2. (a) *Standard Condition*.—Standard condition of jute yarn or rove shall be that reached by the yarn or rove in skein form when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

(b) *Moisture Equilibrium*.—It shall be considered that moisture equilibrium is reached when, after free exposure to air in motion, two successive weighings not less than 4 hr. apart show not more than 0.1 per cent progressive increase in weight. Moisture equilibrium shall be

approached from the dry side (not moisture-free).

(c) *Direction of Twist*.—The direction of twist shall be as defined in the Standard Definitions of Terms Relating to Textile Materials (A.S.T.M. Designation: D 123) of the American Society for Testing Materials, as follows:

*Twist, Direction of*.—A yarn has **S** twist if, when held in a vertical position, the spirals conform in direction of slope to the central portion of the letter “S,” and **Z** twist if the spirals conform in direction of slope to the central portion of the letter “Z.”



S Twist

Z Twist

(d) *Yarn or Rove Number*.—The number of a single jute yarn or jute rove

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1942.



shall be the weight in pounds of 14,400 yd. (one spyndle) of yarn or rove.

(e) *Tolerances*.—Tolerances are the limits within which the specified characteristics of the yarn or rove must come to constitute good delivery on contract.

## TOLERANCES

### Breaking Strength

3. The average breaking strength of the yarn or rove shall be not less than the specified breaking strength.

### Yarn or Rove Number

4. The average yarn or rove number shall be as specified within the following permissible variations:

Untreated yarn or rove . . . . .	$\pm 5$ per cent
Treated yarn or rove, including impregnant . . . . .	$\pm 10$ per cent

### Twist

5. (a) *Direction*.—The direction of twist shall be **S** or **Z**, as specified.

(b) *Turns*.—The average turns per inch of yarn or rove shall be as specified, within the following permissible variations:

Single rove . . . . .	$\pm 10$ per cent
Ply twist in yarn or rove . . . . .	$\pm 10$ per cent

### Ethylene Dichloride Extractable Matter

6. (a) The average percentage extractable matter in the untreated yarn or rove shall not vary from the specified content by more than plus or minus 2 per cent of the conditioned weight of the sample.

(b) The average percentage extractable matter in yarn or rove treated with pitch, asphalt, tar, or similar material, shall not vary from the specified content by more than plus or minus 20 per cent. For other treatments the tolerances on extractable matter shall be as specified.

## Precision

7. The tolerances for yarn number, twist, and extractable matter are intended to represent the maximum deviation from the true average of the lot to be expected in sampling, using the number of tests specified, and assuming a probability of 9 chances in 10, as indicated by information available at the present time.

NOTE.—Additional information will be collected by the committee, and should other tolerances be indicated, changes will be made accordingly.

## METHODS OF TESTING

### Test Conditions

8. The yarn or rove shall be brought to standard condition in accordance with Section 2 (a) and (b) prior to testing, and all tests for yarn number, twist, and breaking strength, also the weighing of samples for determination of extractable matter shall be made under standard atmospheric conditions.

### Sampling

9. From each shipment made in one lot, ranging from ten bales to 40,000 lb., one unit for testing shall be taken at random from each of ten bales. From shipments of less than ten bales, ten units shall be taken, with at least one unit from each bale.

NOTE.—A unit is the package as used by the consumer, such as a ball, tube, reel, cone, skein, etc. A test specimen is the portion of a unit upon which the test is actually performed.

### Test Specimens

10. (a) *Yarn or Rove 500 ft. per lb. and Over*.—A test specimen consisting of one 30-yd. skein shall be prepared from each test unit using any reel having an accurately measured perimeter not less than  $1\frac{1}{2}$  yd. and which distributes the strands so that there is no overlapping.

(b) *Yarn or Rove Under 500 ft. per lb.*—A test specimen consisting of two accurately measured lengths each about 14.4 yd. in length shall be taken from each test unit, by measuring along a flat surface.

(c) Care shall be exercised to prevent stretching or loss of twist of the material.

### Breaking Strength

11. (a) *Preparation.*—All tests for breaking strength shall be made on single strands. In making the tests care shall be taken to prevent any change in twist of the material. When the specimens have been prepared in skein form (Section 10 (a)), the single strand shall be drawn from the skein mounted on a suitable type of collapsible reel. When specimens have been prepared by flat measurement (Section 10 (b)), special care shall be taken to avoid loss of twist.

(b) *Testing Machine.*—All single strands shall be broken on a pendulum type of power-driven testing machine or other equivalent approved type of apparatus, of suitable capacity. Pendulum type machines should preferably be of such capacity that, at the breaking point, the pendulum makes an angle between 9 and 45 deg. with the vertical. The dial or scale shall be divided into units not exceeding those given in the following table:

Breaking Load, lb.	Divisions, lb.
Under 25 .....	0.1
25 to 100, excl. ....	0.5
100 and over .....	2.0

(c) *Procedure.*—Specimens shall be held between drums, capstans, or approved type of clamps. The distance between centers or contact points of the drums, capstans, or clamps shall be not less than 20 nor more than 24 in. The pulling speed of pendulum type machines shall be  $12 \pm \frac{1}{2}$  in. per min. All breaks within  $\frac{1}{2}$  in. of the contact points shall be discarded.

(d) *Number of Tests.*—At least five acceptable breaks (Paragraph (c)) shall be made on each specimen tested. The average breaking strength shall be the arithmetic mean of the total number of tests.

### Yarn or Rove Number

12. The weight of each conditioned skein or length of yarn or rove shall be determined on a balance accurate to within 0.25 per cent of the weight of the specimen. The weight per spindly shall then be calculated in pounds to the nearest 0.1 lb., or in feet per pound to the nearest 0.1 ft. The yarn or rove number or the feet per pound shall be the arithmetic mean of all results.

### Twist

13. (a) *Removal of Impregnant.*—The impregnant shall be removed before determination of twist, using proper precautions to prevent changes of twist during extraction.

(b) *Direction of Twist.*—The direction of twist shall be determined in accordance with Section 2 (c).

(c) *Procedure.*—Provided no change in twist has occurred, skeins or lengths remaining from other tests may be used. Otherwise, new test specimens shall be prepared, in accordance with Section 10 (a) or (b). The turns per inch of twist in the material shall be determined on any approved twist tester with clamps placed 10 in. apart. The total number of turns required to untwist divided by 10 gives the turns per inch.

(d) *Number of Tests.*—Twist test shall be made on at least three test specimens taken not less than 3 yd. apart from each of five or more skeins or test lengths. The twist shall be the arithmetic mean of the results of 15 or more tests.

### Ethylene Dichloride Extractable Matter

14. (a) *Test Specimens.*—Five 20-g. specimens (approximate) shall be taken

from each of five of the conditioned units representing a shipment and the arithmetic average of the results obtained on these 25 specimens, calculated in accordance with Paragraph (d), shall be the percentage oily and fatty substances in the shipment.

(b) *Conditioned Weight*.—Each conditioned test specimen shall be weighed separately to the nearest 0.025 g. to obtain the weight of the conditioned test specimen, weight *A*.

(c) *Procedure*.—Each test specimen (Paragraph (a)) shall be extracted separately in a Soxhlet apparatus, until the liquid in the extraction tube is clear, siphoning not less than 20 times with ethylene dichloride (boiling point 82.5 to 84 C., acidity less than 0.001 per cent) (Note 1). The extraction apparatus (Note 2) shall be of sufficient capacity to hold the specimen easily. The opening in the extraction tube shall be protected by a filter free from extractable matter, such as a filter thimble, a wrapping of filter paper, or a thin layer of cotton or glass wool. The solvent shall then be evaporated from the extract, the residue dried at 100 to 105 C., and cooled to room temperature in a desiccator. The residue shall then be weighed to the nearest 1 mg. The residue shall be returned to the oven and the drying, cooling, and weighing shall be repeated until there is no progressive change in weight. The residue consists of oils, fats, waxes, pitch, asphalt, tar, and components of the natural fiber extractable by the solvent. The dry weight of the extract is weight *B*.

NOTE 1.—Carbon tetrachloride, c.p., ethyl ether, U.S.P., benzol, c.p., or any petroleum fraction distilling below 90 C. may be substituted for the ethylene dichloride.

NOTE 2.—The type of extraction apparatus described in the Standard Methods of Testing

and Tolerances for Knit Goods (A.S.T.M. Designation: D 231) of the American Society for Testing Materials<sup>3</sup> may be used.

(d) *Calculations*.—The content of oily and fatty substances shall be calculated as a percentage of the original conditioned weight of the specimen by means of the following formula:

$$E = \frac{B \times 100}{A}$$

where:

*E* = percentage of oily and fatty substances (conditioned basis),

*A* = weight of conditioned specimen (Paragraph (b)), and

*B* = weight of dry extract (Paragraph (c)).

#### Total Loading Content

15. Total sizing and finishing materials may be approximated on fresh conditioned specimens by means of the procedure outlined in Section 5 (c) and (e) of the Tentative Methods of Quantitative Analysis of Textiles (A.S.T.M. Designation: D 629) of the American Society for Testing Materials.<sup>3</sup> This method is not accurate for impregnants containing latex, some resins, and certain plastics, and it does not correct for the normal soluble material of natural fiber.

#### Retests

16. Whenever the results of tests indicate average values varying from the specified values by amounts in excess of permissible variations, check tests shall be made in accordance with these methods.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

# *Tentative Method of Test for*

## COMPATABILITY OF GLASS YARN WITH INSULATING VARNISH<sup>1</sup>



A.S.T.M. Designation: D 886 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method of test applies to the quantitative evaluation of the compatibility of glass yarns with insulating varnish, that is, how well the varnish wets the yarn, and to the indication of the effect of sizing on the impregnation of the yarn by the varnish.

(b) The method involves the following general operations: winding multiple layers of glass yarn on a copper wire, immersing the covered wire in varnish, and determining the amount of varnish retained by each successive layer of yarn by removing and weighing each layer of yarn separately.

### Test Specimens

2. Ten test specimens shall be prepared in accordance with Paragraphs (a) to (c).

(a) Wrap eight layers of No. 450 $\frac{1}{2}$ , 12 ends, glass yarn on copper wire having a diameter of 0.162 in. by winding each

successive layer in the reverse direction using moderate tension, and with approximately 11 turns per inch, taking care to avoid skips or piling of the yarn.

NOTE.—A properly wrapped wire should show an increase of  $0.038 \pm 0.005$  in. over the diameter of the bare wire when measured, preferably by microprojection, or by a microscope equipped with a micrometer eyepiece.

(b) Using shellac cut with alcohol, apply a band, approximately 0.5 in. in width at 10-in. intervals along the length of the insulated wire (Paragraph (a)). Allow the shellac to dry and then cut the wire into ten 10-in. lengths through the center of the band of shellac which will prevent raveling of the yarn.

(c) The test specimen (Paragraph (b)) shall be dried for 4 hr. at 105 to 110 C., placed in a desiccator and allowed to cool to room temperature, and then treated in accordance with Section 4.

### Standard Varnish

3. (a) The standard varnish<sup>3</sup> contain-

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-13 on Textile Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> The finished varnish can be obtained from the Irvington Varnish and Insulator Co., Irvington, N. J. by using the order number RL 5829. The body viscosity of 10 cm. is measured by a fluid meter of their design.

ing the following ingredients shall be prepared as described in Paragraph (b):

Ingredients	
Dehydrated castor oil, GH .....	7 lb., 13 oz.
Pentalyn G.....	3 lb., 15 oz.
Dehydrated castor oil, GH .....	7 lb., 13 oz.
Mineral spirits.....	3 gal.
Manganese nuolate, 4 per cent.....	33 g.
Lead nuolate, 16 per cent.....	87 g.

(b) Mix the dehydrated castor oil GH and Pentalyn G and heat to 550 F., then add the dehydrated castor oil and heat at 580 to 590 F., until a heavy body (10-cm. viscosity) develops. Cool to 325 F., thin, and add the driers. This varnish shall contain 50 per cent solids by weight, and shall have a viscosity of 150 centipoises at 75 F.

#### Procedure

4. (a) *Varnish Impregnation.*—The test specimens shall be immersed in the standard varnish for 30 min. by standing them vertically in a 250-ml. glass graduate so that they are completely covered with varnish. The specimens shall then be removed from the varnish, allowed to drain in a vertical position for 30 min., and then inverted and baked in an oven at 290 F. for 20 min. (Note).

NOTE.—This treatment will remove the excess varnish solvent without baking the specimens too hard.

(b) A 4-in. section shall be cut from the center of each of the baked specimens and each layer of yarn from each specimen shall be unwrapped and weighed to the nearest 1 mg., weight *A*. Each unwrapped layer of yarn shall be placed in a ventilated oven and heated for 1 hr. at a temperature of  $500 \pm 20$  C., cooled in a desiccator to room temperature, and then weighed to the nearest 1 mg., weight *B*.

#### Sizing

5. The sizing on the unvarnished glass yarn shall be determined in its "as received" condition in accordance with

the following procedure: Five test specimens, each weighing not less than 5 g., shall be dried for 4 hr. at 105 to 110 C., allowed to cool to room temperature in a desiccator, and then weighed to the nearest 1 mg. weight *C*. The specimens shall then be placed in a ventilated oven and heated for 1 hr. at a temperature of  $500 \pm 20$  C., cooled in a desiccator to room temperature, and then weighed to the nearest 1 mg., weight *D*.

#### Calculations

6. The percentages of sizing, coating (varnish and sizing), and of varnish without sizing shall be calculated as follows:

$$I = \frac{A - B}{A} \times 100$$

$$S = \frac{C - D}{C} \times 100$$

$$V = I - S$$

where:

*I* = percentage of coating (varnish and sizing),

*S* = percentage of original sizing,

*V* = percentage of varnish,

*A* = weight of layer of yarn after baking (Section 4(b)),

*B* = weight of layer of yarn after ignition (Section 4(b)),

*C* = weight of "as received" yarn specimen before ignition (Section 5), and

*D* = weight of the "as received" yarn specimen after ignition (Section 5).

#### Report

7. The following shall be reported for each layer of varnished glass yarn:

(1) Weight of coated specimen before and after ignition,

(2) Weight of coating,

(3) Percentage of coating,

(4) Percentage of sizing, and

(5) Percentage of varnish.

# *Tentative Methods of*

## TESTING VARNISHED GLASS FABRICS AND VARNISHED GLASS FABRIC TAPES USED IN ELECTRICAL INSULATION<sup>1</sup>



**A.S.T.M. Designation: D 902 - 46 T**

*(Formerly Part of Methods D 295)*

ISSUED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover procedures for the testing of varnished glass fabrics and varnished glass fabric tapes (Note) to be used as electrical insulation.

NOTE.—Methods of testing varnished cloths and tapes are given in the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295).<sup>3</sup>

### SAMPLING

#### Selecting Rolls

2. (a) Shipments of varnished glass fabric and varnished glass fabric tapes shall be sampled as specified in Paragraphs (b) and (c). The rolls or pads shall be selected in such a manner as to be representative of the shipment.

(b) *Fabric*.—One roll shall be selected from each ten rolls or fraction thereof in a shipment of full-width fabric.

(c) *Tape*.—Rolls of tape shall be selected from each shipment in accordance with the following schedule:

Number of Rolls in Shipment	Minimum Number of Sample Rolls
Over 10 000.....	1 per thousand
5 001 to 10 000.....	10
2 001 to 5 000.....	5
501 to 2 000.....	2
200 to 500.....	1
Less than 200.....	optional

#### Selecting Samples

3. (a) Except for tape shipped packed in oil, not less than two turns of fabric or six turns of tape from each roll or pad selected for sampling shall be cut off and discarded before the samples are selected. The number of samples required for purpose of tests shall then be cut across the entire width of each roll or pad. The length of the samples shall be not less than 36 in. (91 cm.).

(b) From shipments such as sheets of fabric or strips of tape, the samples

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials.

<sup>2</sup> Accepted by the Administrative Committee on Standards, December 31, 1946.

These methods were formerly included in the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (D 295 - 44 T), but were withdrawn from Methods D 295 in 1946, revised, and issued under the present designation.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall be representative of the shipment and shall be taken in accordance with Paragraph (a).

### CONDITIONING

#### Conditioning

4. (a) If it is desired to test the material in the condition as received by the purchaser, the test samples shall not be specially conditioned, but the packages containing the rolls of fabric or tape from which the samples are to be taken shall be allowed to reach approximately test room temperature before the packages are opened and the samples cut. Specimens to be tested shall be removed from the roll as required and tested immediately, unless otherwise specified.

(b) Where it is desired to test in a controlled atmosphere, the test specimens shall be conditioned for 48 hr. in air maintained at a temperature between 68 and 86 F. (20 and 30 C.) and at a relative humidity of  $50 \pm 2$  per cent.

(c) If a conditioning cabinet or chamber is used, test specimens shall be subjected to test immediately upon withdrawal from the cabinet or chamber, unless otherwise specified.

### THICKNESS

#### Test Specimens

5. Test specimens shall consist of samples selected in accordance with Section 3.

#### Procedure

6. The thickness (Note) shall be measured in accordance with Method B of the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374),<sup>3</sup> modified as follows:

(1) In making thickness measurements, only one layer of material shall be used.

(2) In the case of fabrics, ten measurements equally spaced across the width of the specimen shall be taken and the thickness of the fabric shall be the average of the ten measurements.

(3) In the case of tapes, unless otherwise specified, ten measurements equally spaced along the length of each specimen shall be taken and the thickness of the tape shall be the average of the ten measurements.

NOTE.—The average thickness, as obtained for the as-received condition dielectric strength test (Section 13), may be conveniently used instead of the separate test described in Section 6.

#### Report

7. The report shall include the average, maximum, and minimum thicknesses in inches.

### BREAKING STRENGTH

#### Apparatus

8. (a) A testing machine<sup>4</sup> of the dead-weight pendulum type shall be used. The machine preferably shall be power driven.

(b) The capacity of the machine shall not exceed 500 lb. (227 kg.).

(c) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing specimens breaking at 50 lb. (22.7 kg.) or over, and to 0.5 lb. or 0.5 kg. or less for testing specimens breaking under 50 lb. (22.7 kg.)

#### Test Specimens

9. (a) Specimens cut from the full width fabric samples or from sample rolls of tapes over 1 in. (2.54 cm.) in width shall be 1 in. (2.54 cm.) in width (Note) and shall have lengths not less than 2 in. (5.08 cm.) in excess of the jaw clearance of the testing machine.

<sup>4</sup> This apparatus is similar to the apparatus used for the testing of paper, which is described in Section 10 of the Tentative Methods of Sampling and Testing Untreated Paper Used in Electrical Insulation (A.S.T.M. Designation: D 202), see p. 685.



For tape having a nominal width of 1 in. (2.54 cm.) or under, the specimens shall be of the original width (Note) and shall have lengths not less than 2 in. (5.08 cm.) in excess of the jaw clearance of the testing machine.

NOTE.—In the case of specimens 1 in. (2.54 cm.) in width and having ultimate breaking loads above the capacity of the machine, it is permissible to reduce the width of the specimen to 0.5 in. (1.27 cm.).

(b) In the case of fabrics, five specimens shall be cut with the sides parallel to the warp threads and five with the sides parallel to the filling threads from samples selected in accordance with Section 3.

(c) In the case of tapes, five specimens shall be cut from each roll selected in accordance with Section 2 (c).

### Procedure

10. (a) The ratio of the clearance distance between jaws to the width of the specimen shall be not less than 5 to 1 nor more than 10 to 1.

(b) The rate of travel of the movable jaw shall be constant and preferably 12 in. (30.5 cm.) per min., but it may be within the limits of 11 and 13 in. (28 and 33 cm.) per min., provided it is constant.

(c) All readings obtained when the specimen breaks at or in the jaws shall be rejected.

### Report

11. (a) The breaking strength of a roll of fabric or tape shall be the average of the breaking strengths of all the specimens tested from the roll. The report shall include the average, maximum, and minimum breaking strengths expressed in pounds per inch width or kilograms per centimeter width, together with the width and the nominal thickness.

(b) In the case of fabrics, the breaking strength of the warp threads and the filling threads shall be reported separately.

## DIELECTRIC STRENGTH

### *Short-Time and Step-by-Step Tests*

#### Purpose

12. The purpose of this test is to determine the dielectric strength of varnished glass fabrics and varnished glass fabric tapes, when subjected to a short-time or step-by-step test.

#### Dielectric Strength

13. (a) The dielectric strength shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> except as specified in the following Sections 14 to 19.

(b) Dielectric strength tests may be applied to specimens of the material in the as-received condition, to specimens from tests for resistance to oil, or to specimens that have been given humidity conditioning.

NOTE.—Work is now under way to develop a suitable test method for conducting dielectric strength tests at elevated temperatures (175 C.).

#### Apparatus

14. For the testing of narrow specimens, as prescribed in Section 15, it is necessary to use a special form of testing device having electrodes  $\frac{1}{4}$  in. (0.64 cm.), whereby the test specimen shall be clamped under pressure in order to prevent flashover around the edges of the material. Two forms of these testers are described in the Appendix to the Tentative Methods of Testing Varnished Cloths and Varnished Cloth Tapes Used in Electrical Insulation (A.S.T.M. Designation: D 295).<sup>3</sup>

## Test Specimens

15. (a) In the case of fabrics, the specimens shall be cut across the full width of each sample selected in accordance with Section 3 and cut in the form of a piece of tape at least 1 in. (2.54 cm.) in width.

(b) In the case of tapes, the specimens shall be cut from samples selected in accordance with Section 3.

## Surrounding Medium

16. Tests shall be made in air unless flashover is encountered, in which case the material may be tested under oil.

## Conditioning

17. Specimens shall be conditioned in accordance with Section 4.

## Procedure

18. (a) Tests shall be made by the short-time test method, except that those tests to be conducted on specimens tested in the as-received condition shall be made by either the short-time test or the step-by-step test, or by both methods.

(b) In the short-time test, voltage shall be increased at the rate of 0.5 kv. per sec.

(c) In tests made by the step-by-step test, each step shall be of 20-sec. duration. Voltage shall be increased by the following increments:

Nominal Thickness  
of Tape

8 mils or less.....	250 v.
Over 8 mils.....	500 v.

The starting voltage shall be adjusted to the nearest even 250 or 500 v., depending on the increment of increase. The starting voltage should preferably be based upon the breakdown voltage in the short-time test, as provided in A.S.T.M. Methods D 149. Where this may not be feasible or convenient, a starting voltage may be selected as the result of test experience, of such a

value that at least three testing steps are obtained prior to breakdown.

## Report

19. The report shall include the following:

(1) The test method used for determining the dielectric strength,

(2) The average, maximum, and minimum dielectric strength in volts per mil,

(3) The number of shots for each test method,

(4) The temperature at which the test was made,

(5) The conditioning treatment, and

(6) The value of the initially applied voltage in the step-by-step test.

## POWER FACTOR AND DIELECTRIC CONSTANT

### Purpose

20. The purpose of this test is to determine the power factor and dielectric constant of varnished glass fabrics and varnished glass fabric tapes at frequencies not exceeding 70 cycles per second.

### Definitions and Theory of Test

21. The definitions of dielectric power factor, dielectric constant, and related terms, and the theory of test shall be as given in the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

### Electrodes

22. Flat, rigid, guarded electrodes, not over 10 sq. in. in area, of such size as to give the bridge sufficient sensitivity to detect readily a change in power factor of 0.0005, shall be used. The specimen shall extend beyond the guard electrode for a distance of at least four times the thickness of the specimen. The pressure shall be not less than 10 nor more than 20 psi.

### Conditioning

23. The test specimens shall be conditioned by one of the following two methods:

(a) Where tests in the as-received condition are desired, or where an approximation to some conditions of use is desired, the test specimens shall be conditioned in accordance with Section 4.

(b) When greater reproducibility of results is desired, the test specimens in one thickness shall be heated to  $105 \pm 3$  C. for 1 hr. without vacuum and then for 2 hr. at  $105 \pm 3$  C. in a vacuum having an absolute pressure not exceeding 1 mm. of mercury.

### Procedure

24. (a) Tests shall be made at a temperature of  $25 \pm 3$  C. and at  $125 \pm 1$  C. for oleoresinous coatings, while for silicone varnished glass fabrics the test temperatures shall be  $25 \pm 3$  C. and  $175 \pm 1$  C. The conditioned specimens shall be tested in single thicknesses within 5 min. after removal from the roll or vacuum container. The voltage gradient shall be 50 v. per mil. The method of measurement shall conform to that described in A.S.T.M. Methods D 150.

(b) Three specimens shall be tested in single thicknesses.

### Report

25. The report shall include the following:

(1) *Test Conditions.* — The frequency in cycles per second, temperature in degrees Centigrade, relative humidity in per cent, the conditioning of the specimen, size of electrodes, pressure on electrodes, and voltage gradient in the dielectric while under test.

(2) *Test Specimen.* — Capacitance of specimen in micro-microfarads, effective area of specimen electrodes, and

average thickness of specimen between electrodes.

(3) Power factor, dielectric constant, and loss factor of each specimen, and the average.

(4) Method of measurement.

### RESISTANCE TO OIL

#### Purpose

26. The purpose of this test is to determine the effect of oil on the varnish film and dielectric strength of varnished glass fabrics and varnished glass fabric tapes.

#### Test Specimens

27. One specimen 12 in. (30.5 cm.) in length and not exceeding 1.5 in. (3.8 cm.) in width shall be cut from each sample selected in accordance with Section 3.

#### Procedure

28. (a) In the case of oleoresinous coatings the varnished glass specimens shall be immersed for 15 min. in an insulating oil maintained at a temperature of  $100 \pm 3$  C. (Note). The oil used in this test shall be an insulating oil unless otherwise specified, having a flash point of not less than 135 C. (275 F.) as determined by the Cleveland Open Cup in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>5</sup> In the case of silicone varnished fabrics a lubricating oil shall be used.

NOTE.—Work is now under way to determine the immersion time and temperature as well as the type of lubricating oil for this test.

(b) At the end of the period of immersion the specimens shall be removed from the oil, allowed to cool for at least 30 min. to room temperature, and any excess oil removed by placing the specimens between blotters without any sliding.

## Examination

29. The varnish film shall be examined for disintegration in the oil and flaking either in the oil or on the blotter. Disintegration in the oil may be detected by examination of the used oil for turbidity.

NOTE.—The oil may be considered turbid if a sample of used oil filtered through filter paper is distinctly less transparent than an unfiltered sample of the unused oil when the two samples, in identical containers, are held in front of a diffused light. Flaking along the cut edges of tapes shall not be considered as disintegration of the varnish film.

## Dielectric Strength

30. Specimens which have been allowed to cool at least 30 min. may be tested for dielectric strength any time within a period of 4 hr. after removal from the oil. Five puncturing voltages shall be taken on the specimens in accordance with Section 13, using only the short-time test described in A.S.T.M. Methods D 149. The dielectric strength shall be calculated on the basis of the average thickness of the specimen determined immediately before the dielectric breakdown test.

## Report

31. The report shall include the following:

(1) The type of oil used (preferably including the flash point determined in accordance with the Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92)),<sup>5</sup>

(2) The temperature of the oil,

(3) Results of the physical examination of the film (Section 29),

(4) The percentage increase in average thickness due to oil test, and

(5) The average, maximum, and minimum dielectric strength in volts per mil after the oil tests.

## WEIGHT

### Procedure

32. Specimens of sufficient size to weigh not less than 5 g. (0.18 oz.) shall be accurately weighed on an analytical balance and the weight shall be reported on some commercial unit basis such as the weight per square yard, the weight per gross yard per 1 in. in width, or the weight per roll.

## THREADS PER INCH

### Procedure

33. The count or threads per inch shall be determined separately on the warp and filling by counting in a space of not less than 1 in. in at least five different places in the sample strip (or strips). The average of the five determinations shall be the count.

### Report

34. The results of the warp count and the filling count shall be reported separately as threads per inch.

NOTE 1.—The warp threads in fabrics are the threads that are parallel with the length dimension.

NOTE 2.—Before counting black varnished materials it will be necessary to remove the varnish film with a knife blade or other suitable instrument. As an alternate method, liquid varnish removers may be used for this purpose, provided specimens are dried before the thread count is taken.

## HEAT RESISTANCE

NOTE.—A test method for determining heat resistance is now under consideration.

## *Tentative Specifications for*

### ENCLOSURES FOR SMALL TESTING MACHINES FOR TESTS AT SUBNORMAL AND SUPERNORMAL TEMPERATURES OF ELECTRICAL INSULATING MATERIALS AND PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 760 - 44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. These specifications cover chambers suitable for total enclosure of small testing machines, such as are required in A.S.T.M. methods of testing electrical insulating materials and plastics. They are designed for use when tests are to be made below or above normal temperatures, specifically within the range from  $-70$  to  $+170$  F.

NOTE 1.—Enclosures of the type covered by these specifications are suitable for use in connection with the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256),<sup>3</sup> Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48),<sup>3</sup> Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621),<sup>3</sup> and Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

NOTE 2.—These enclosures for small testing machines are not preferred to a room of the walk-in type, but have been found satisfactory for high-temperature and low-temperature tests of electrical insulating materials and plastics

where the expense, mechanical problems, and other known disadvantages of a complete room are not justified.

#### Requirements

2. The enclosures shall be of double-walled or equivalent construction with a dead air space or suitable insulation between the walls. They shall be in the form of a box having inside dimensions 14 in. in width by 48 in. in length by 38 in. in height. They either shall be open on the bottom for convenient placing over bench-mounted testing machines or have one side removable to admit the testing machine. Each enclosure shall be provided with a double-walled sliding door 10 by 12 in. fitted with a window through which the scale of the testing machine may be read. A suitable hole for connecting ducts shall be located at the center of one end of the box, 14 in. from the bottom.

#### Materials of Construction

3. The enclosures may be constructed of transparent or opaque plastic, plywood, pressed wood or pressed pulp, or pressed asbestos board. If constructed of a material which is moisture-sensitive, they should be coated with a suitable moistureproof paint, varnish, or lacquer.

<sup>1</sup>Under the standardization procedure of the Society, these specifications are under the joint jurisdiction of A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup>Accepted by Committee E-10 on Standards, February 8, 1944.

<sup>3</sup>Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## *Tentative Specifications for*

### SERVICING UNITS FOR TESTS AT SUBNORMAL AND SUPERNORMAL TEMPERATURES OF ELECTRICAL INSULATING MATERIALS AND PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 761 - 44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. These specifications cover servicing units for maintaining atmospheres in suitable enclosures to which they are attached, for tests of electrical insulating materials and plastics at subnormal and supernormal temperatures, at any desired temperature within the range from -70 F. to +170 F.

NOTE.—These servicing units and associated chambers are not preferred to a room of the walk-in type, but have been found satisfactory for high-temperature and low-temperature tests of electrical insulating materials and plastics where the expense, mechanical problems, and other known disadvantages of a complete room are not justified.

#### Requirements

2. The individual servicing units shall be of a size and shape which will make them easily portable, and means shall be available for connecting the units to test enclosures. They shall conform to the detailed requirements specified in Sections 3 to 7.

#### Housing

3. The servicing unit shall be housed in a suitable double-walled insulated box, having a layer of insulation composed of a minimum of 2 in. of asbestos, glass wool, or equivalent, between the two walls. Both inner and outer walls shall be coated with a suitable moisture-resistant and rust-resistant paint, enamel, or lacquer.

#### Blower and Duct System

4. A blower system shall provide a minimum of 40 cu. ft. per min. of air. It shall be provided with a by-pass damper which shall function to maintain the temperature at any given point by means of a control located within the attached enclosure.

#### Heating Capacity

5. Each servicing unit shall be equipped with fin-type electric heaters having a minimum rating of 1600 w.

#### Refrigeration

6. Cooling of the unit shall be accomplished either by means of mechanical

<sup>1</sup>Under the standardization procedure of the Society, these specifications are under the joint jurisdiction of A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup>Accepted by Committee E-10 on Standards, February 8, 1944.

refrigeration or through the use of dry ice. In the latter case, a removable basket, capable of holding 50 lb. of dry ice, shall be provided.

#### Temperature Control

7. Each servicing unit shall be provided with thermostatic temperature control which may be coupled either through a switch or a double outlet box,

to permit operation of the damper in the range  $-70$  to  $+50$  F. without the heating elements. The bulb for the thermostat shall have a 48-in. flexible extension so that it may be placed at the desired point in the enclosure. The complete apparatus shall be sensitive to plus or minus 2 F. throughout the entire range of  $-70$  to  $+170$  F.



# *Tentative Specifications for* PHENOLIC MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 700 - 45 T

ISSUED, 1943; REVISED, 1945.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover hot-molding thermosetting compounds consisting of essentially a phenol-formaldehyde resin either alone or intimately combined in the uncured or partially cured condition with fillers, pigments and dyes, as required, to obtain the properties desired.

NOTE.—The properties included in these specifications are those required to identify the types of molding materials covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types

2. These specifications cover ten types of phenolic molding compounds classified in accordance with their filler, and

electrical, mechanical, and heat-resisting characteristics, as follows:

*Type 1.*—An unfilled transparent or translucent material suitable for both electrical and mechanical applications (Note 1).

*Type 2.*—General purpose wood-flour-filled material suitable for both electrical and mechanical applications (Note 1).

*Type 3.*—General purpose material with a cellulose filler which may be wood flour or cotton flock. It is somewhat higher in impact strength than type 2 and suitable for both mechanical and electrical applications (Note 1).

*Type 4.*—Moderate impact strength material with cotton rag or other suitable forms of cellulose filler to give the required strength for this type (Notes 1 and 2).

*Type 5.*—Medium impact strength material with cotton rag or other suitable forms of cellulose filler to obtain the required strength for this type (Notes 1 and 2).

*Type 6.*—High impact strength material with cotton rag or other suitable

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by Administrative Committee on Standards, December, 12, 1945.

forms of cellulose filler to obtain the required strength for this type (Note 2).

*Type 7.*—An electrical high-frequency low-loss material with a mineral filler (Note 3).

*Type 8.*—A superior electrical high-frequency low-loss material with a mineral filler (Note 3).

*Type 9.*—General purpose mineral-filled material for both electrical and mechanical applications (Note 1).

*Type 10.*—Heat-resistant mineral-filled material (Note 4).

**NOTE 1.**—To secure the optimum electrical qualities inherent in these materials, they must be dried to remove surplus moisture prior to molding. Powder should be spread in shallow nonmetallic pans preferably in a rotating oven. Preformed material should be supported in such a way as to heat evenly. Material for test specimens must be dried immediately prior to molding and molded while still warm. Warming for 30 min. at 185 F. usually suffices, or shorter times at higher temperatures may be used, although more care may be required to prevent precuring the material.

**NOTE 2.**—There will be some sacrifice in appearance and moldability.

**NOTE 3.**—The powder must be dried before molding as described in Note 1.

**NOTE 4.**—The physical properties of this type of material may change by about 10 per cent when exposed continuously for 7 days at a maximum temperature of 400 F.

### General Requirements

3. (a) The molding compounds shall consist of essentially a phenol-formaldehyde resin of such characteristics and combined in the uncured or partially cured state in such proportion and in such manner with the filler as to flow under heat and pressure to fill the mold completely and to harden so as to hold metal inserts firmly.

(b) The particle size, pourability, apparent density, bulk factor, flow (plasticity), color, and obtainable finish shall be as specified by the purchaser in the contract or order.

**NOTE.**—Molding powders and compounds are usually supplied meeting the following values for the various properties:

Type	Particle Size <sup>a</sup>	Pourability, <sup>a</sup> max., sec.	Apparent Density, <sup>a</sup> min., g per cu cm.	Bulk Factor, <sup>a</sup> max.
	100 per cent Passing A.S.T.M. Sieve			
1.....	No. 14	30	0.50	2.5
2.....	No. 10	25	0.50	3.0
3.....	No. 10	...	0.40	3.5
4.....	...	...	0.20	10
5.....	...	...	0.08	18
6.....	...	...	...	18
7.....	No. 12	35	0.65	3.0
8.....	No. 12	35	0.65	3.0
9.....	...	...	...	12.5
10.....	No. 12	25	0.70	2.8

<sup>a</sup> Methods for determination of particle size, pourability, apparent density, and bulk factor are given in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup>

### Detail Requirements

4. The various types of molding compounds shall have such properties that when molded by compression under optimum molding conditions in accordance with the provisions of the Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48) of the American Society for Testing Materials,<sup>3</sup> the test specimens will conform to the requirements as to physical and electrical properties prescribed in Table I.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

**Methods of Testing**

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—For those tests where conditioning is required, the molded test specimens of phenol-formaldehyde resin shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials.<sup>3</sup>

Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Flexural Strength.*—Tentative Methods D 48<sup>3</sup> and Tentative Method of Flexural Test of Plastics (A.S.T.M. Designation: D 790).<sup>3</sup>

(f) *Impact Strength (Izod).*—Tentative Methods D 48<sup>3</sup> and Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup>

(g) *Tensile Strength.*—Tentative Methods D 48<sup>3</sup> and Tentative Method of Test for Tensile Strength of Molded

TABLE I.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

	Type 1	Type 2	Type 3	Type 4	Type 5	Type 6	Type 7	Type 8	Type 9	Type 10
Water absorption, max. weight gain, per cent.....	0.20	0.80	0.80	1.50	1.75	1.75	0.10	0.07	0.20	0.20
Specific gravity, 25/25 C. (77/77 F.), max.	1.30	1.45	1.45	1.45	1.45	1.45	2.00	2.00	1.95	2.00
Flexural strength, min., psi.....	12 000	9 000	9 000	9 000	9 000	9 000	8 000	8 000	9 000	7 500
Impact strength (Izod), min., ft.-lb. per in. of notch.....	0.30	0.24	0.34	0.80	1.75	4.00	0.30	0.30	0.64	0.25
Tensile strength ( $\frac{1}{8}$ -in. specimen), min., psi.....	7 000	6 500	6 500	5 500	6 000	6 000	4 500	4 500	4 500	4 500
Compressive strength, min., psi.....	22 000	22 000	22 000	18 000	15 000	15 000	15 000	15 000	15 000	15 000
Mold shrinkage, in. per in. (min.).....	0.010	0.006	0.005	0.002	0.002	0.003	0.001	0.001	0.001	0.002
..... (max.).....	0.014	0.008	0.008	0.007	0.006	0.006	0.004	0.004	0.005	0.006
Insulation resistance, min., megohms.....	100	100	100	100	100	100	300	300	300	300
Dielectric strength, (Short-time test.....	300	275	240	200	200	200	350	350	150	250
min., v. per mil. (Step-by-step test.....	...	225	200	75	75	50	300	300	75	200
Dielectric constant, 60 cycles.....	...	...	...	...	...	...	6.0	6.0	...	...
max.....	...	...	...	...	...	...	5.0	5.0	...	...
Power factor, max., 60 cycles.....	...	...	...	...	...	...	0.030	0.025	...	...
1,000,000 cycles.....	...	...	...	...	...	...	0.015	0.009	...	...
Loss factor, max., 60 cycles.....	...	...	...	...	...	...	0.180	0.150	...	...
1,000,000 cycles.....	...	...	...	...	...	...	0.075	0.045	...	...

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(c) *Water Absorption.*—Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48)<sup>3</sup> and Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion.

(d) *Specific Gravity.*—Method A of the Tentative Methods of Test for Specific

Electrical Insulating Materials (A.S.T.M. Designation: D 651),<sup>3</sup> using the  $\frac{1}{8}$ -in. test specimen.

(h) *Compressive Strength.*—Tentative Methods D 48<sup>3</sup> and Tentative Method of Test for Compressive Strength of Plastics (A.S.T.M. Designation: D 695).<sup>3</sup>

(i) *Shrinkage.*—Standard Method of Measuring Shrinkage from Mold Dimensions of Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 551).<sup>3</sup>

(j) *Insulation Resistance.*—Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials

(A.S.T.M. Designation: D 257),<sup>3</sup> using the test specimen and electrodes shown in Fig. 2.

(k) *Dielectric Strength*.—Tentative Methods D 48<sup>3</sup> and Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> except that the test specimens for both short-time and step-by-step tests shall be conditioned for 48 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, and tested under these same conditions.

(l) *Dielectric Constant, Power Factor, and Loss Factor*.—Tentative Methods D 48<sup>3</sup> and Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150),<sup>3</sup> except that the test specimens shall be conditioned for 48 hr. at  $50 \pm 0.5$  C. ( $122 \pm 1$  F.), then cooled in a desiccator over anhydrous calcium chloride to the temperature of the room in which the test will be made. Application of electrodes and completion of measurements shall then be made within a maximum period of 15 min. after removal from the desiccator.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that

routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Tentative Specifications for*

## CELLULOSE NITRATE (PYROXYLIN) PLASTIC SHEETS, RODS, AND TUBES<sup>1</sup>



A.S.T.M. Designation: D 701 - 46 T

ISSUED, 1943; REVISED, 1944, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover sheets, rods, and tubes made of cellulose nitrate (pyroxylin) plastics, plasticizers, and stabilizers, pigments and dyes, as required.

NOTE.—The properties included in these specifications are those required to identify the types and grades of materials covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Type

2. These specifications cover sheets, rods, and tubes made of the following four types of cellulose nitrate plastic:

*Type I.*—General purpose, transparent,

*Type II.*—Improved toughness, transparent,

*Type III.*—General purpose, pigmented, and

*Type IV.*—Improved toughness, pigmented.

### General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The size, shape, dimensional tolerances, surface finish, and color shall be as specified by the purchaser in the contract or order.

### Detail Requirements

4. (a) *Cellulose Nitrate.*—The cellulose nitrate used in the production of cellulose nitrate plastic shall have a nitrogen content of 10.65 to 11.25 per cent. It shall be thoroughly purified and shall have an A.S.T.M. stability at 134.5 C. (275 F.) of not less than 30 min.

(b) *Composition.*—Cellulose nitrate plastic shall be manufactured from approved cellulose nitrate (Paragraph (a)) with suitable plasticizers, or from reworked material having an acidity not greater than 25 epm. (Note 1, Section 6) and a fuming-off temperature of not less

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

than 165 C. (329 F.) (Section 6 (c)), or from mixtures of the two. It may contain dye-stuffs, pigments, or stabilizers as required.

(c) *Residual Volatile Matter*.—The residual volatile matter shall be such that the material will not warp or buckle excessively on aging under normal room conditions. Residual volatile matter for

form in which they are to be shipped (Note), sufficient to determine conformance of the material to these specifications.

NOTE.—The phrase "the form in which they are to be shipped" is used to designate material having the type of finish specified by the purchaser in the contract or order, since various finishing methods can have an effect on the properties.

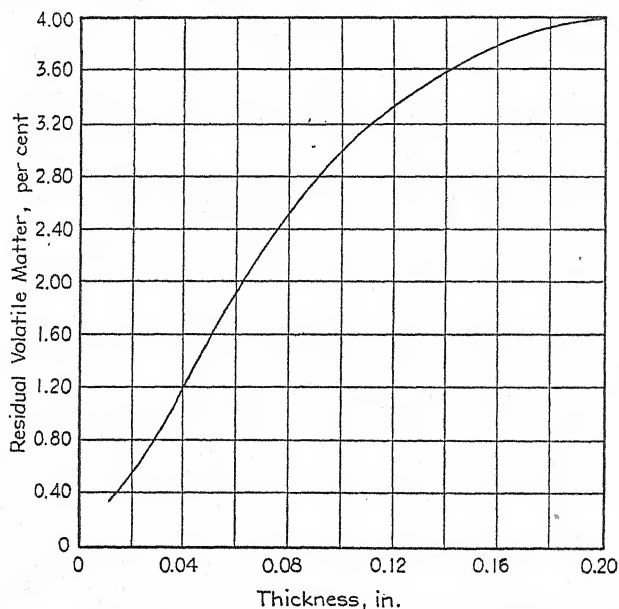


FIG. 1.—Maximum Residual Volatile Matter Permissible for Various Thicknesses of Cellulose Nitrate Plastic.

each thickness specified shall not be more than the amounts shown in Fig. 1.

(d) *Fuming-off Temperature*.—Cellulose nitrate plastic shall have a fuming-off temperature of not less than 165 C. (329 F.).

(e) *Acidity*.—The free acidity of the finished plastic shall not exceed 15 epm. (Note 1, Section 6).

(f) The material shall conform to the requirements as to physical properties prescribed in Table I.

#### Sampling

5. A sample shall be selected from the finished sheets, rods, and tubes, in the

#### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Nitrogen Content and Stability*.—Standard Specifications and Tests for Soluble Nitrocellulose (A.S.T.M. Designation: D 301).<sup>3</sup>

(b) *Residual Volatile Matter*.<sup>4</sup>—From the sample, cut shavings 0.005 to 0.010 in.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part II.

<sup>4</sup> This method of test is intended as a temporary procedure for use until an A.S.T.M. tentative method, now being developed in Committee D-20 on Plastics, has been completed.

in thickness, transferring these shavings immediately into a tared weighing bottle until approximately 1 g. of the sample has been prepared. Care shall be taken to keep evaporation of solvents at a minimum. Weigh the sample and the container to the nearest 0.1 mg. Heat for 2 hr., with the cover removed, in a vacuum oven at 50 C. (122 F.) under a vacuum of at least 28 in. of mercury. Cool, weigh, and return to the oven for another 2 hr. at 50 C. (122 F.). Cool and weigh again. The loss in weight

these tests are used, the vacuum oven test for determining residual volatile matter, as described in this paragraph, shall be used as referee in case of dispute. It must be further recognized that the vacuum oven test provides relative data only; it does not measure the absolute value of the volatile constituents.

(c) *Fuming-off Temperature.*<sup>4</sup>—The sample shall be prepared by taking shavings not thicker than 0.001 in. from the test piece, which shall be clean and free of finger-prints. The shavings shall

TABLE I.—DETAIL REQUIREMENTS FOR CELLULOSE NITRATE PLASTIC SHEETS.

		Type 1, General Transparent				Type 2, Improved Toughness, Transparent				Type 3, Pig- mented, General Opaque				Type 4, Pig- mented, Improved Toughness, Opaque			
		0.003-0.029 in.	0.030-0.059 in.	0.060-0.125 in.	Over 0.125 in.	0.003-0.029 in.	0.030-0.059 in.	0.060-0.125 in.	Over 0.125 in.	0.003-0.029 in.	0.030-0.059 in.	0.060-0.125 in.	Over 0.125 in.	0.003-0.029 in.	0.030-0.059 in.	0.060-0.125 in.	Over 0.125 in.
Specific gravity,																	
25/25 C. (77/77 F.)	{ min. ....	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35	1.35
	{ max. ....	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.40	1.57	1.57	1.57	1.57	1.57	1.57	1.57	1.57
Tensile strength, min.,	psi. ....	8000	7000	6000	5000	6000	5000	4000	3000	6500	5500	4500	3500	7000	6000	5000	4000
Elongation, min., per	cent. ....	25	25	25	25	35	35	35	35	10	10	10	10	20	20	20	20
Impact strength	{ at 25 C. (77 F.) ....			3.0	3.0			4.0	4.0			1.0	1.0			2.0	2.0
(Izod), min., ft.-lb.	{																
per in. of notch	{ at -40 C. (-40 F.) ....			0.75	0.75			1.0	1.0			0.25	0.25			0.50	0.50
Hardness, Rockwell	{ max. ....			120	120							120	120				
(R-scale)	{ min. ....			90	90							90	90				
Haze value (no color),	max., per cent. ....	2.0	3.0	4.0	8.0	2.0	3.0	4.0	8.0								
Water absorption,	{ Weight gain plus																
(24-hr. immersion)	{ soluble matter loss. ....	3.5	3.0	2.5	2.0	4.0	3.5	3.0	2.5	3.5	3.0	2.5	2.0	4.0	3.5	3.0	2.5
max., per cent	{ Soluble matter loss	2.0	1.5	1.0	0.5	2.5	2.0	1.5	1.0	2.0	1.5	1.0	0.5	2.5	2.0	1.5	1.0
Heat distortion tem-	{ deg. Cent. ....			60	60			49	49			60	60			49	49
perature at 264 psi.	{ deg. Fahr. ....			140	140			120	120			140	140			120	120
fiber stress, min.																	

during the second heating is assumed to be due to the loss of plasticizer, and loss of plasticizer is also assumed to take place at the same rate during the first heating period. Calculate the residual volatile matter as follows:

$$\text{Residual volatile matter, per cent} = \frac{\text{first wt. loss} - \text{second wt. loss}}{\text{wt. of sample}} \times 100$$

It is recognized that there are other methods of test which are more efficient as control tests but which are of limited application and cannot be used in a general specification. In case any of

be taken by scraping the plastic with a suitable scraper or with a freshly broken piece of glass. To free the shavings of excessive moisture and solvents, they shall be heated for 2 hr. in an oven at 50 C. (122 F.). Approximately 0.1 g. of the blended shavings shall be introduced into a dry, clean, 6 by 0.5-in. test tube, which shall then be *lightly* stoppered with a new perforated or notched cork stopper and immersed 2 in. in a liquid bath at 100 C. (212 F.). The bath shall be heated so that the temperature rises at the rate of not less than 3 C. (5 F.) nor more than 5 C. (9 F.) per min. until



the sample decomposes explosively. This temperature shall be recorded as the fuming-off temperature. Not fewer than five tests shall be run on each sample, and the average of the five reported as the fuming-off temperature. The test shall be repeated for all samples that do not decompose explosively.

(d) *Free Acid*.—A  $1.00 \pm 0.1$ -g. sample of the material shall be dissolved in 10 ml. of distilled water and 100 ml. of acetone. When solution is complete, the cellulose nitrate and plasticizers shall be precipitated by adding another 25 ml. of water in such a manner that a fine slurry is formed. A few drops of methyl red indicator shall be added and the suspension titrated with 0.01 *N* sodium hydroxide. A blank shall be run on the acetone-water mixture. Calculate the amount of free acid as follows:

$$\text{Acidity, epm.} = \frac{(R - S)N}{W} \times 1000$$

where:

*R* = milliliters of NaOH solution required to titrate the sample,

*S* = milliliters of NaOH solution required to titrate the blank,

*N* = normality of the NaOH solution, and

*W* = grams of sample used.

NOTE 1.—Epm. is an abbreviation for equivalents per million. It is a useful measure for expressing acidity or alkalinity in terms of acidic hydrogen concentration in parts per million. Acidity expressed as epm. is independent of the kind of acid present.

NOTE 2.—In case a dye in the plastic obscures the methyl red end point, the end point may be taken at a pH of 5.3 in electrometric titration.

(e) *Conditioning Test Specimens*.—For those tests where conditioning is required, the test specimens of cellulose nitrate plastic sheets, rods, and tubes shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and

Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>5</sup>

(f) *Test Conditions*.—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(g) *Specific Gravity*.—Method A of Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>5</sup>

(h) *Tensile Strength and Elongation at Rupture*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>5</sup>

(i) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>5</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>5</sup>

(j) *Rockwell Hardness*.—Tentative Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 785),<sup>5</sup> using the R scale.

(k) *Haze Value*.—Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell (A.S.T.M. Designation: D 672).<sup>5</sup>

(l) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>5</sup> using the 24-hr. immersion procedure.

(m) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>5</sup> using a fiber stress of 264 psi.

## Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Sec-

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tion 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### **Retest and Rejection**

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these

two additional sets of specimens fails, the batch of molding material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### **Packing and Marking**

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Tentative Specifications for*

## POLYSTYRENE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 703 - 44 T

ISSUED, 1943; REVISED, 1943, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover thermoplastic polystyrene molding compounds for use particularly in the injection molding process.

NOTE.—The properties included in these specifications are those required to identify the types of molding compounds covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Types

2. These specifications cover the following three types of polystyrene molding compounds:

*Type 1.*—A general purpose compound consisting of an unmodified form of polystyrene (polyvinyl benzene) with or without the addition of pigments, colors, and lubricants as may be required.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, January 1, 1944.

*Type 2.*—A superior electrical purpose compound consisting of an unmodified form of polystyrene (polyvinyl benzene) which may contain small amounts of lubricants intended to facilitate removal of molded objects from the mold.

*Type 3.*—A general purpose molding material characterized by superior heat-resistant qualities.

### General Requirements

3. (a) The molding compound shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The particle size (Note) and color shall be as specified by the purchaser in the contract or order.

NOTE.—Polystyrene molding compounds are generally supplied in a granular size of the following sieve analysis:

Retained on $\frac{3}{8}$ -in. sieve . . . . .	none
Passing No. 14 (1410-micron) sieve . . . . .	not over 6 per cent

(c) The material covered by type 3 of these specifications shall consist of polystyrene compound, with halogenated polynuclear aromatic hydrocarbons of the type and quantity necessary to confer the specified properties.

## Detail Requirements

4. (a) *Molding Compounds*.—The compounds covered by these specifications shall conform to the following requirements:

	Type 1	Type 2	Type 3
Methanol soluble content, max., per cent.....	3.5	3.0	...
Viscosity of 10 per cent solution in toluene, min., centipoises.....	30	40	...

(b) *Molded Test Specimens*.—Test specimens molded by injection under conditions specified by the manufacturer for both types of compounds shall conform to the requirements as to physical and electrical properties prescribed in Table I.

TABLE I.—DETAIL REQUIREMENTS FOR INJECTION MOLDED TEST SPECIMENS.

	Type 1	Type 2	Type 3
Specific gravity, 25/25 C. (77/77 F.).....	1.07	1.40	1.30
Refractive index $n_D$ .....	1.05	1.05	1.30
Impact strength (Izod), min., ft.-lb. per in. of notch.....	1.600	1.600	...
Heat distortion temperature at 264 psi. fiber stress, min.....	1.585	1.585	...
Deformation under load, max., per cent.....	0.30	0.30	0.25
Power factor, { 60 cycles.....	76 C.	76 C.	84 C.
max. { 1,000 cycles.....	169 F.	169 F.	183 F.
Dielectric constant { 60 cycles.....	1.5	1.5	1.5
max. { 1,000 cycles.....	0.0005	0.0003	0.002
max. { 1,000,000 cycles.....	0.0005	0.0003	0.003
Dielectric constant { 60 cycles.....	0.0010	0.0005	0.001
max. { 1,000 cycles.....	2.50	2.50	2.60
max. { 1,000,000 cycles.....	2.75	2.60	2.80
max. { 1,000,000 cycles.....	2.50	2.50	2.60
max. { 1,000,000 cycles.....	2.75	2.60	2.80

## Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

## Methods of Testing

6. The properties enumerated in these

specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens*.—For those tests where conditioning is required, the molded test specimens of polystyrene shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions*.—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(c) *Sieve Analysis*.—Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392).<sup>3</sup>

(d) *Methanol Solubility Test*.—The methanol soluble content shall be determined as follows:

(1) *SAMPLE PREPARATION*.—Grind the sample to pass an A.S.T.M. No. 12 (1680-micron) sieve.

(2) *APPARATUS*.—The following apparatus will be required: Analytical balance, 50-ml. beakers with watch glasses (or weighing bottles), 400-ml. beakers with watch glasses, 25- and 200-ml. graduated cylinders, dense sintered glass crucibles, source of vacuum, vacuum filter flask, desiccator, water bath or steam plate, drying oven adjustable to 65 to 70 C. (149 to 158 F.), crucible tongs, and tweezers.

(3) *SOLVENTS*.—Redistilled c. p. methanol (Note 1), c. p. dioxane, toluene, and c. p. acetone will be required as solvents.

(4) *PROCEDURE*.—Wash all glassware with toluene, follow with an acetone rinse, and dry thoroughly. Dry the

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

crucibles for 1 hr. at 65 C. (149 F.) and store in the desiccator. Weigh directly into a tared 50-ml. beaker (or weighing bottle) and weigh to the nearest 0.0001 g. about 0.3 g. of the ground sample. Add 15 ml. of dioxane (Note 2), cover with a watch glass, and let stand until dissolved. After solution is complete, stir thoroughly and transfer rapidly, while stirring, to a 400-ml. beaker containing 200 to 250 ml. of redistilled methanol. Rinse the 50-ml. beaker with 25 to 50 ml. of methanol from a wash bottle, using a policeman if necessary to dislodge any solid particles. The transfer shall be quantitative.

Heat to 65 C. (149 F.) on the water bath or steam plate, while stirring, until the precipitate is fairly well coagulated, then remove and allow to settle for a few minutes. Filter with suction through a weighed sintered glass crucible (Note 3), first decanting as much liquid as possible, and then transferring the solids with the aid of a policeman and methanol from a wash bottle. Use about 125 ml. of methanol for transferring and washing the precipitate. Continue suction until dry. Clean the outside of the crucible with a damp cloth or chamois. Dry to constant weight at 65 to 70 C. (149 to 158 F.) (Note 4). Cool in the desiccator, and weigh.

(5) CALCULATION.—Calculate the percentage methanol soluble content as follows:

$$\text{Methanol soluble content, per cent} = \frac{A - B}{A} \times 100$$

where:

$A$  = weight of sample in grams, and  
 $B$  = weight of precipitate in grams.

NOTE 1.—The methanol shall be redistilled before initial use, and may be recovered by redistillation and reused two or three times.

NOTE 2.—C. p. benzene (thiophene free), or methylethylketone may be substituted for the dioxane in routine analyses.

NOTE 3.—The strictest quantitative technique shall be observed in handling the sample and the crucible.

NOTE 4.—The drying time may vary from 1 to 3 hr. and may be divided conveniently into equal periods with a weighing between each. If a weight change greater than 0.4 mg. occurs, an additional heating for 1 hr. is usually sufficient to reach constant weight. For routine analyses the precipitate may be dried for 1 hr. at 100 C. (212 F.); the crucible in this case should first be dried at 100 C. (212 F.) instead of 65 C. (149 F.).

(e) *Viscosity of 10 per cent Solution.*—The viscosity of a 10 per cent solution by weight of polystyrene in toluene shall be determined as follows:

(1) APPARATUS.—A 16-oz. screw cap bottle, platform type balance accurate to 0.01 g., coarse fritted glass filter, thermometer, and shaking or tumbling device will be required.

(2) TOLUENE.—The toluene shall conform to the Standard Specifications for Industrial Grade Toluene for Use in Paint, Varnish, Lacquer, and Related Products (A.S.T.M. Designation: D 362).<sup>4</sup>

(3) SAMPLE PREPARATION.—Grind the sample to pass an A.S.T.M. No. 12 (1680-micron) sieve.

(4) PREPARATION OF SOLUTION.—Prepare a 10 per cent solution by weight of polystyrene in toluene as follows: Weigh to the nearest 0.01 g. the sample and the toluene, respectively, into a clean 16-oz. small-mouth screw-cap bottle. The final volume in the bottle shall be not more than 50 per cent of the capacity of the bottle. Cap tightly, and tumble or shake until all the sample is dissolved. Adjust the temperature of the solution to  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.) and determine the specific gravity of the solution at 25/25 C. by the Standard Method of Test for Specific Gravity of Road Oils, Road Tars, Asphalt Cement, and Soft Tar Pitches (A.S.T.M. Designation: D 70).<sup>4</sup>

(5) **PROCEDURE.**—Filter the solution through the coarse fritted glass filter and determine the viscosity of the solution at  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.) in accordance with the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445),<sup>4</sup> using Method B in the Appendix of Method D 445 and the modified Ostwald Viscosimeter (Note 6). Select a pipette suitable for the approximate viscosity and calibrate it with a standard viscosity sample at 25 C. (77 F.). Convert the viscosity in terms of centistokes to absolute viscosity in centipoises by the following equation (Note 7):

$$\text{Absolute viscosity in centipoises} = V \times s$$

where:

$V$  = kinematic viscosity in centistokes, and

$s$  = specific gravity of the solution.

**NOTE 5.**—The 10 per cent solution shall be free of undissolved particles or lint. The transfer of solution shall be done rapidly to avoid evaporation of solvent. The apparatus must be cleaned immediately after use by passing through successive portions of toluene, benzene or methylethylketone, and acetone, after which dry air may be used to evaporate the acetone.

**NOTE 6.**—For routine analysis, the calibrated Hoeppler or Ubbelohde viscosimeters may be used as described in Appendix A of the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445),<sup>4</sup> or the viscosity may be determined in accordance with the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>4</sup>

**NOTE 7.**—This equation may be used to convert centistokes to centipoises regardless of the method used in determining the viscosity. If the Saybolt Universal Viscosimeter is used, convert seconds efflux to centipoises by the following equation:

Absolute Viscosity in centipoises =

$$100 \times s \times \left[ (0.0022 \times t) - \frac{1.8}{t} \right]$$

where:

$s$  = specific gravity of the solution, and

$t$  = efflux time in seconds, Saybolt Universal viscosity.

(f) **Specific Gravity.**—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(g) **Refractive Index.**—Standard Methods of Test for Index of Refraction of Transparent Organic Plastics (A.S.T.M. Designation: D 542).<sup>3</sup>

(h) **Impact Strength (Izod).**—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256),<sup>3</sup> except that the notch shall be produced by a machining operation.

(i) **Heat Distortion Temperature.**—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using a fiber stress of 264 psi.

(j) **Deformation Under Load.**—Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621).<sup>3</sup>

(k) **Power Factor and Dielectric Constant.**—Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150).<sup>3</sup>

#### Number of Tests<sup>5</sup>

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

<sup>5</sup> Editorially revised in June, 1944.

**Retest and Rejection**

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from

the receipt of the material by the purchaser.

**Packing and Marking**

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.



# *Tentative Specifications for*

## MELAMINE-FORMALDEHYDE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 704 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover thermo-setting molding compounds consisting of a melamine-formaldehyde resin binder, with or without small amounts of other resins, that has been intimately combined in the uncured or partially cured condition with fillers, pigments, and other chemical agents.

NOTE.—The properties included in these specifications are those required to identify the types of molding compounds covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Types

2. These specifications cover the following four types of melamine-formaldehyde molding compounds:

*Type 1.*—Melamine-formaldehyde with alpha cellulose filler.

*Type 2.*—Melamine-formaldehyde with mineral filler.

*Type 3.*—Melamine-formaldehyde with chopped cotton-rag filler.

*Type 4.*—Melamine formaldehyde-phenol formaldehyde (85:15 ratio) with chopped cotton-rag filler.

### General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The apparent density, bulk factor, flow, particle size, and color of the compound shall be as specified by the purchaser in the contract or order.

### Detail Requirements

4. Test specimens molded by compression under conditions specified by the manufacturer shall conform to the requirements as to physical and electrical properties prescribed in Table I.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392).<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

Used for Electrical Insulation (A.S.T.M. Designation: D 48).<sup>3</sup>

(e) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256),<sup>3</sup> except that the test specimen shall be built up of four pieces each  $\frac{1}{8}$  by  $\frac{1}{2}$  in. in cross-section.

TABLE I.—DETAIL REQUIREMENTS FOR COMPRESSION MOLDED TEST SPECIMENS.

	Type 1	Type 2	Type 3	Type 4
Specific gravity, 25/25 C. (77/77 F.) {min.....	1.45	1.70	1.45	1.45
.....{max.....	1.55	2.20	1.55	1.55
Flexural strength, min., psi.....	9 000	7500	10 500	13 000
Impact strength (Izod), min., ft.-lb. per in. of notch.....	0.20	0.25	0.55	0.9
Water absorption (24-hr., immersion), max. weight gain, per cent.....	1.8	0.15	1.0	1.1
Dielectric strength, {short-time test.....	300	300	300	160
min., v. per mil. {step-by-step test.....	250	250	230	125
Arc resistance, min., sec.....	80	120	120	8

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens*.—For those tests where conditioning is required, the molded test specimens of melamine-formaldehyde shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions*.—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the test methods or in these specifications.

(c) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(d) *Flexural Strength*.—Tentative Methods of Testing Molded Materials

(f) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion.

(g) *Dielectric Strength*.—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> except that the test specimens shall be compression molded disks or plates  $\frac{1}{8}$  in. in thickness, conditioned for 48 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.) at  $50 \pm 2$  per cent relative humidity, and tested under the same conditions.

(h) *Arc Resistance*.—Standard Method of Test for Arc Resistance of Solid Electrical Insulating Materials (A.S.T.M. Designation: D 495).<sup>3</sup>

### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish con-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

formity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of the two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser.

Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# Tentative Specifications for CELLULOSE ACETATE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 706 - 46 T

ISSUED, 1943; REVISED, 1943, 1944, JUNE 1946, SEPTEMBER 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover thermoplastic molding compounds consisting of cellulose acetate and plasticizers, with or without the addition of dyes and pigments, and suitable for compression, injection, or extrusion molding. They do not include special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the types and grades of molding compounds covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types and Grades

2. These specifications cover four types and fifteen grades of cellulose acetate molding compounds, classified in accordance with their flow temperatures as shown in Table I.

tate molding compounds, classified in accordance with their flow temperatures as shown in Table I.

TABLE I.—TYPES AND GRADES OF CELLULOSE ACETATE MOLDING COMPOUNDS.

Type	Grade	Flow Temperature	
		deg. Fahr.	deg. Cent.
I. General purpose	1	314 ± 9	156 ± 5
	2	300 ± 9	149 ± 5
	3	286 ± 9	141 ± 5
	4	272 ± 9	133 ± 5
II. Heat resistant	5	356 ± 9	180 ± 5
	6	332 ± 9	167 ± 5
	7	314 ± 9	156 ± 5
III. Impact resist- ant.....	8	300 ± 9	149 ± 5
	9	286 ± 9	141 ± 5
	10	272 ± 9	133 ± 5
	11	260 ± 9	127 ± 5
IV. Moisture resist- ant.....	12	332 ± 9	167 ± 5
	13	314 ± 9	156 ± 5
	14	300 ± 9	149 ± 5
	15	286 ± 9	141 ± 5

## General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The size and form of granulations of the molding compound (Note), and for color, translucency, or opacity shall be as specified by the purchaser in the contract or order.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revisions accepted by the Society at annual meeting, June, 1946, and by the Administrative Committee on Standards, September 9, 1946.

NOTE.—Cellulose acetate molding compounds are available in granular sizes of  $\frac{1}{8}$ ,  $\frac{1}{16}$ , and  $\frac{1}{32}$  in., and in other sizes and forms.

### Detail Requirements

4. Test specimens molded by compression or injection under conditions specified by the manufacturer shall conform to the requirements as to physical and electrical properties prescribed in Table II.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard

(a) *Conditioning Test Specimens.*—For those tests where conditioning is required, the molded test specimens of cellulose acetate shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the test method or in these specifications.

TABLE II.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

		Type I, General Purpose				Type II, Heat Resistant			Type III, Impact Resistant				Type IV, Moisture Resistant			
		Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6	Grade 7	Grade 8	Grade 9	Grade 10	Grade 11	Grade 12	Grade 13	Grade 14	Grade 15
Flow temperature.....	(deg. Cent. $\pm 5$ C. .... deg. Fahr. $\pm 9$ F. ....)	156	149	141	133	180	167	156	149	141	133	127	167	156	149	141
Specific gravity, 25/25 C. (77/77 F.), max.....		314	300	286	272	356	332	314	300	286	272	260	332	314	300	286
Tensile strength, min., at 25 C. (77 F.).....		1.34	1.34	1.34	1.34	1.34	1.34	1.34	1.32	1.32	1.32	1.32	1.34	1.34	1.34	1.34
Tensile strength, min., at 71 C. (160 F.).....		5200	4300	3500	2900	6200	5500	5000	4000	3500	3000	2500	5600	5000	4100	3300
Heat distortion temperature at 264 psi fiber stress, min., deg. Cent.....		2800	2000	1400	1100	4000	2700	2300	2000	1400	900	600	3000	2400	1700	1200
Heat distortion temperature at 264 psi fiber stress, min., deg. Fahr.....		63	54	52	46	74	68	63	52	49	43	38	66	60	52	46
Impact strength (Izod), min., ft.-lb. per in. of notch, at 25 C. (77 F.).....		0.8	1.0	1.3	1.7	0.5	0.5	0.8	1.7	1.9	2.2	3.0	0.9	1.1	1.4	2.0
Impact strength (Izod), min., ft.-lb. per in. of notch, at -40 C. (-40 F.).....		0.3	0.3	0.4	0.4	0.3	0.3	0.3	0.4	0.4	0.4	0.5	0.4	0.4	0.4	0.4
Water absorption (24-hr. immersion), max., per cent, (Weight gain plus soluble matter loss.....)		4.0	3.5	3.7	4.0	5.0	5.0	4.7	3.8	4.5	5.5	6.0	2.9	2.7	2.8	3.2
Weight loss on heating (72 hr. at 82 C. (180 F.)), max., per cent.....		0.5	0.7	0.9	1.1	0.3	0.3	0.3	0.8	1.0	1.2	1.5	0.2	0.3	0.7	1.3
		3.0	4.5	5.5	7.5	0.75	1.5	2.3	5.6	7.1	9.3	11.3	1.6	2.5	4.3	6.2

Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(c) *Flow Temperature.*—Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (A.S.T.M. Designation: D 569),<sup>3</sup> using the preferred method of conditioning.

(d) *Specific Gravity.*—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Tensile Strength.*—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638)<sup>3</sup> and the Tentative Methods of Test for Tensile and Compressive Properties of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation:

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

D 759).<sup>3</sup> Conditioning time at 71 C. (160 F.) shall be not less than 1 hr. nor more than 2 hr.

(f) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using a fiber stress of 264 psi.

(g) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Super-normal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

(h) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion.

(i) *Weight Loss on Heating*.—Test specimens 3 by 1 by  $\frac{1}{8}$  in. shall be conditioned for 48 hr. over anhydrous calcium chloride at  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall be weighed and then placed in a circulation air oven for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.). The specimens shall be supported flatwise on a screen in the oven. Upon removal from the oven, the specimens shall be cooled in a desiccator over anhydrous calcium chloride to  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall then be weighed, and the percentage weight loss on heating calculated on the basis of the conditioned weight.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in

Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer and the number of the contract or order.

# Tentative Specifications for CELLULOSE ACETATE BUTYRATE MOLDING COMPOUNDS<sup>1</sup>



**A.S.T.M. Designation: D 707 - 46 T**

**ISSUED, 1943; REVISED, 1943, 1944, 1946.<sup>2</sup>**

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover thermoplastic molding compounds consisting of cellulose acetate butyrate having a butyryl content of not less than 35 per cent, and plasticizers, with or without the addition of dyes and pigments, and suitable for compression, injection, and extrusion molding.

**NOTE.**—The properties included in these specifications are those required to identify the types of molding material covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types

2. These specifications cover three types and ten grades of cellulose acetate butyrate molding compounds, classified

in accordance with their flow temperatures, as shown in Table I.

**TABLE I.—TYPES OF CELLULOSE ACETATE BUTYRATE MOLDING COMPOUNDS.**

Type	Grade	Flow Temperature	
		deg. Fahr.	deg. Cent.
I. General purpose	1....	300 ± 9	149 ± 5
	2....	286 ± 9	141 ± 5
	3....	300 ± 9	149 ± 5
	4....	286 ± 9	141 ± 5
II. Heat resistant...	5....	332 ± 9	167 ± 5
	6....	314 ± 9	156 ± 5
III. Impact resistant	7....	272 ± 9	133 ± 5
	8....	260 ± 9	127 ± 5
	9....	272 ± 9	133 ± 5
	10....	260 ± 9	127 ± 5

## General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The particle size, dimensions, and color shall be as specified by the purchaser in the contract or order (Note).

**NOTE.**—Cellulose acetate butyrate molding compounds are available in the following forms: pellets ( $\frac{1}{8}$  in.), granulations ( $\frac{1}{8}$  to  $\frac{5}{16}$  in.), and

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.



blanks and molding sheets ( $\frac{5}{32}$  to  $1\frac{1}{8}$  in. in thickness with a maximum area of 20 by 25 in.). These compounds are available in the following colors: plain colors (transparent, translucent, opaque, black) variegations (configurations of plain colors), and pearls (variegated with iridescent luster).

### Detail Requirements

4. Test specimens cut or milled from sheets  $\frac{1}{8}$  in. in thickness which have been molded by compression in a positive type mold under conditions specified by the manufacturer shall conform to the re-

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

#### (a) Conditioning Test Specimens.—

For those tests where conditioning is required, the molded test specimens of cellulose acetate butyrate shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical

TABLE II.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

Properties		Type I, General Purpose				Type II, Heat Resistant		Type III, Impact Resistant			
		Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6	Grade 7	Grade 8	Grade 9	Grade 10
Flow temperature.....	(deg. Cent. $\pm 5$ C. .... deg. Fahr. $\pm 9$ F. ....)	149 300	141 286	149 300	141 286	167 332	156 314	133 272	127 260	133 272	127 260
Specific gravity, 25/25 C. (77/77 F.), max. ....		1.21	1.20	1.22	1.21	1.23	1.22	1.19	1.18	1.22	1.21
Tensile strength, min., at 25 C. (77 F.) .....		3500	2700	4100	3600	5300	4500	2000	1400	3100	2500
psi, at 71 C. (160 F.) .....		2100	1300	2200	1600	3600	2800	800	400	1100	600
Impact strength (Izod), at 25 C. (77 F.) .....		1.5	1.8	1.0	1.2	0.6	1.0	2.3	3.1	1.7	2.5
min. ft.-lb. per in. of notch, at -40 C. (-40 F.) .....		0.5	0.6	0.4	0.5	0.3	0.4	0.7	1.0	0.6	0.7
Heat distortion temperature at 264 psi. fiber stress, min. ....	(deg. Cent. .... deg. Fahr. ....)	50 122	46 114	53 127	48 119	64 148	56 134	43 109	39 103	45 113	41 105
Water absorption (24-hr. immersion), max., per cent	Weight gain plus soluble matter loss. ....	2.1	2.1	1.7	1.8	2.3	2.2	2.1	2.1	2.0	2.4
Weight loss on heating (72 hr. at 82 C. (180 F.)), max., per cent .....	Soluble matter loss. ....	0.3	0.4	0.2	0.4	0.2	0.3	0.5	0.6	0.7	1.1
		1.5	2.0	1.9	3.0	0.4	0.8	2.4	3.0	4.5	6.0

quirements as to physical and electrical properties prescribed in Table II.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392).<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(c) *Flow Temperature.*—Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (A.S.T.M. Designation: D 569),<sup>3</sup> except that the test specimen shall be obtained by punching pieces from a molded sheet and piling them to the required height. Test specimens

\* Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall be conditioned by the preferred method.

(d) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Tensile Strength*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638)<sup>3</sup> and the Tentative Methods of Test for Tensile and Compressive Properties of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 759).<sup>3</sup> Conditioning time at 71 C. (160 F.) shall be not less than 1 hr. nor more than 2 hr.

(f) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

(g) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using a fiber stress of 264 psi.

(h) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion.

(i) *Weight Loss on Heating*.—Test specimens 3 by 1 by  $\frac{1}{8}$  in. shall be conditioned for 48 hr. over anhydrous calcium chloride at  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall be weighed and then placed in a circulation air oven for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.). The specimens shall be supported flatwise on a screen in the oven. Upon

removal from the oven, the specimens shall be cooled in a desiccator over anhydrous calcium chloride to  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall then be weighed and the percentage weight loss on heating calculated on the basis of the conditioned weight.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the

manufacturer within three weeks from the receipt of the material by the purchaser.

#### **Packing and Marking**

9 (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest

rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer and the number of the contract or order.

# *Tentative Specifications for* VINYL CHLORIDE-ACETATE RESIN PLASTIC SHEETS<sup>1</sup>



A.S.T.M. Designation: D 708 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover rigid plastic sheets made of vinyl chloride-acetate resins of approximately 90 per cent vinyl chloride content and the necessary lubricants, stabilizers, dyes, pigments and fillers. These specifications do not cover special sheets compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the type of material covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Type and Grades

2. These specifications cover one type and three grades of rigid plastic sheets made from vinyl chloride-acetate resin.

## General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) No sheet shall contain more than eight surface defects over  $\frac{1}{32}$  in. in diameter or equivalent, more than 1 in. from the edge, and of the type described in the Standard Descriptive Nomenclature of Objects Made from Plastics (A.S.T.M. Designation: D 675) of the American Society for Testing Materials.<sup>3</sup> The total defective area or areas shall not exceed 1 per cent of the sheet.

(c) The grade designation, color, surface finish, and size shall be as specified by the purchaser in the contract or order (Notes 1, 2, and 3).

NOTE 1.—Vinyl chloride-acetate resin sheets are available with any of the following finishes:  
Press polished both sides,  
Matte finished both sides, and  
Press polished one side, matte finished one side.

NOTE 2.—Standard trim size for these sheets is  $20 \pm \frac{1}{16}$  in. by  $50 \pm \frac{1}{16}$  in. by the specified thickness.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, August 28, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE 3.—The three grades of sheets covered by these specifications are available in the following degrees of transparency or opacity:

Grade 1.—Transparent clear or colors only,

Grade 2.—Translucent or opaque colors only,  
and

Grade 3.—Opaque colors only.

(d) The maximum thickness of sheets covered by these specifications shall be  $\frac{1}{8}$  in. The average thickness shall be within plus or minus 10 per cent of the specified thickness, but no single thickness measurement shall vary more than 15 per cent from the specified thickness.

plastic composition under similar production conditions.

## Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—

For those tests where conditioning is required, the test specimens formed from rigid sheets of vinyl chloride-acetate resin plastics shall be conditioned in accordance with the Standard Procedure

TABLE I.—DETAIL REQUIREMENTS FOR VINYL CHLORIDE-ACETATE RESIN PLASTIC SHEETS.

	Grade 1	Grade 2	Grade 3
Specific gravity, 25/25 C. {min.....	1.32	1.32	1.40
(77/77 F.) {max.....	1.36	1.60	1.55
Tensile strength, min., psi.....	8 000	6 500	6 000
Modulus of elasticity in flexure, min., psi.....	380 000	350 000	360 000
Impact strength (Izod), min., ft.-lb. per in. of notch.....	0.3	0.35	0.5
Heat distortion temperature at 264 psi. fiber stress, min. {deg. Cent.....	52	57	5½
{deg. Fahr.....	125	135	130
Deformation under load.....	a	a	a
Flammability.....	b	b	b
Index of refraction ( $n_D$ ) {max.....	1.53	.....	.....
{min.....	1.52	.....	.....
Optical displacement factor, max.....	10	.....	.....
Thermal stability, max., mg. of HCl per g.....	2	2	2
Volume resistivity, min., ohm-cm.....	10 <sup>14</sup>	10 <sup>14</sup>	10 <sup>14</sup>
Water absorption (24-hr. immersion), max., per cent.....	0.15	0.15	0.15

<sup>a</sup> To be specified at a later date.

<sup>b</sup> Self-extinguishing.

### Detail Requirements

4. The material shall conform to the requirements as to physical properties prescribed in Table I.

## Sampling

5. A representative sheet shall be taken from each lot of 2000 lb. or fraction thereof and shall be tested to determine conformity with the requirements of these specifications. In addition, sheets may be subjected to individual visual inspection. If the sheets are of such thickness that the necessary test specimens cannot be obtained, the specimens may be cut from a sheet  $\frac{1}{8}$  in. in thickness prepared from the same

of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(c) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(d) *Tensile Strength*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

(e) *Modulus of Elasticity in Flexure*.—Tentative Method of Flexural Test of Plastics (A.S.T.M. Designation: D 790).<sup>3</sup>

(f) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup> The test specimen shall have a milled notch.

(g) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648).<sup>3</sup> using a fiber stress of 264 psi.

(h) *Deformation Under Load*.—Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621).<sup>3</sup>

(i) *Flammability*.—Standard Method of Test for Flammability of Plastics Over 0.050 in. in Thickness (A.S.T.M. Designation: D 635).<sup>3</sup>

(j) *Index of Refraction*.—Standard Methods of Test for Index of Refraction of Transparent Organic Plastics (A.S.T.M. Designation: D 542).<sup>3</sup>

(k) *Optical Displacement*.—Standard Method of Test for Surface Irregularities of Flat Transparent Plastic Sheets (A.S.T.M. Designation: D 637).<sup>3</sup>

(l) *Thermal Stability*.—Tentative Method of Test for Short-Time Stability at Elevated Temperatures of Plastics Containing Chlorine (A.S.T.M. Designation: D 793).<sup>3</sup>

(m) *Volume Resistivity*.—Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257).<sup>3</sup>

(n) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup>

using the 24-hr. immersion procedure and test specimens  $\frac{1}{8}$  in. in thickness.

## Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing the sheet which has been sampled according to Section 5. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

## Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

## Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure ac-

ceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name, stock

number, and surface finish of the material, the size and quantity therein, as defined by the contract or order under which shipment is made, the name of the manufacturer and the number of the contract or order.



# *Tentative Specifications for*

## VINYL CHLORIDE-ACETATE RESIN MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 728 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3. Pa.

### Scope

1. These specifications cover rigid molding compounds consisting of vinyl chloride-acetate resins of approximately 86 per cent vinyl chloride content and the necessary lubricants, stabilizers, dyes, pigments, and fillers, and suitable for injection molding. These specifications do not cover special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the type of molding compound covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Type and Grades

2. These specifications cover one type and three grades of rigid injection molding compounds made from vinyl chloride-acetate resins.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent not assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, August 28, 1944.

### General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The grade designation, color, and particle size of the molding compounds shall be as specified by the purchaser in the contract or order (Notes 1 and 2).

NOTE 1.—Vinyl chloride - acetate resin molding compounds are available in the following particle sizes:

- (1) Chips approximately  $\frac{3}{4}$  by  $\frac{3}{4}$  by  $\frac{1}{8}$  in.,
- (2) All particles passing a  $\frac{3}{16}$ -in. round-hole sieve, and
- (3) All particles passing a  $\frac{5}{16}$ -in. round-hole sieve.

NOTE 2.—The three grades of molding compounds covered by these specifications are available in the following degrees of transparency or opacity:

- Grade 1.—Transparent clear or colors only,
- Grade 2.—Translucent or opaque colors only, and
- Grade 3.—Opaque colors only.

### Detail Requirements

4. Test specimens molded under conditions specified by the manufacturer shall conform to the requirements as to physical properties prescribed in Table I.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in

and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the test method or in these specifications.

(c) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(d) *Tensile Strength*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

(e) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resistance of

TABLE I.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

	Grade 1	Grade 2	Grade 3
Specific gravity, 25/25 C. (77/77 F.) {min.....	1.30	1.30	1.30
max.....	1.35	1.40	1.40
Tensile strength, min., psi.....	7 500	4 500	5 000
Impact strength (Izod), min., {at 25 C. (77 F.).....	0.25	0.35	0.35
ft.-lb. per in. of notch {at -40 C. (-40 F.).....	a	a	a
Heat distortion temperature at 264 psi. fiber stress, min. {deg. Cent.....	52	52	54
{deg. Fahr.....	125	125	130
Deformation under load, max., per cent.....	a	a	a
Flammability.....	b	b	b
Thermal stability, max., mg. HCl per g.....	2	2	2
Volume resistivity, min., ohm-cm.....	10 <sup>14</sup>	10 <sup>14</sup>	10 <sup>14</sup>
Water absorption (24-hr. immersion), max. weight gain, per cent.....	0.15	0.15	0.15

<sup>a</sup> To be specified at a later date.

<sup>b</sup> Self-extinguishing.

accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens*.—For those tests where conditioning is required, the molded test specimens of vinyl chloride-acetate resin plastics shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions*.—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.).

Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup> The test specimen  $\frac{1}{2}$  in. in thickness shall consist of four end-gated, injection-molded pieces  $\frac{1}{8}$  in. in thickness clamped together.

(f) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using an end-gated, injection-molded specimen 5 by  $\frac{1}{2}$  by  $\frac{1}{8}$  in., and using a fiber stress of 264 psi.

(g) *Deformation Under Load*.—Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621).<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(h) *Flammability*.—Standard Method of Test for Flammability of Plastics Over 0.050 in. in Thickness (A.S.T.M. Designation: D 635).<sup>3</sup>

(i) *Thermal Stability*.—Tentative Method of Test for Short-Time Stability at Elevated Temperatures of Plastics Containing Chlorine (A.S.T.M. Designation: D 793).<sup>3</sup>

(j) *Volume Resistivity*.—Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257),<sup>3</sup> using a compression-molded test specimen.

(k) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion procedure and a compression-molded test specimen  $\frac{1}{8}$  in. in thickness.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be

required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name and stock number of the material, size, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer and the number of the contract or order.

# *Tentative Specifications for*

## VINYLDENE CHLORIDE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 729 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover thermoplastic molding compounds composed of a copolymer of vinyl chloride and vinylidene chloride in the approximate ratio of 10 to 90, with suitable plasticizers, stabilizers, dyes, and pigments. The molding compounds are suitable for compression, injection, or extrusion molding.

NOTE.—The properties included in these specifications are those required to identify the types of molding materials covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Type and Forms

2. These specifications cover one general-purpose type of vinylidene chloride material in the form of powder or pellets.

### General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The form and color shall be as specified by the purchaser in the contract or order.

NOTE.—Vinylidene chloride molding compounds are generally supplied in powder and pellet forms conforming to the following sieve analysis:

Powder:	
retained on a No. 18 (1000 - micron) sieve .....	not over 1 per cent
Pellets:	
passing a No. 4 (4760 - micron) sieve .....	100 per cent

### Detail Requirements

4. (a) *Molding Compound*.—The compound covered by these specifications shall conform to the following requirement:

Viscosity of 2 per cent solution in orthodichlorobenzene at 120 C. (248 F.), min., centipoises .....	0.96
--	------

(b) *Molded Test Specimens*.—Test specimens molded by injection under conditions specified by the manufacturer shall conform to the following requirements as to physical and electrical properties:

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent not assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revisions accepted by Committee E-10 on Standards January 1, 1944, and by the Society at annual meeting, June, 1944.

Flow temperature.....	{min.... 132 C. (270 F.) max.... 152 C. (305 F.)}
Specific gravity,	{min..... 1.68 25/25 C. (77/77 F.)... {max..... 1.75
Tensile strength, min., psi.....	4000
Heat distortion temperature at 264 psi. fiber stress, min. ....	66 C. (150 F.)
Water absorption (24-hr. immersion), max. weight gain plus soluble matter loss, per cent. ....	0.1
Dielectric strength, {short-time test..... 350 min., v. per mil. {step-by-step test.... 300	
Flammability.....	self-extinguishing
Impact strength (Izod), min., ft.-lb. per in. of notch.....	{at 25 C. ( 77 F.) 0.5 at -40 C. (-40 F.) 0.1
Weight loss on heating (72 hr. at 82 C. (180 F.)), max., per cent.....	2.0

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392) of the American Society for Testing Materials.<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—Molded test specimens of vinylidene chloride are not sensitive to humidity but shall be conditioned for at least 48 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.) prior to testing, unless otherwise specified in the testing methods or in these specifications.

(b) *Test Conditions.*—Tests shall be conducted in a laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.), unless other-

wise specified in the testing methods or in these specifications.

(c) *Viscosity of 2 per cent Solution.*—The viscosity of the 2 per cent solution shall be determined as follows:

(1) *APPARATUS.*—The apparatus shall consist of the following: Series 50 modified Ostwald viscosimeter conforming to the requirements prescribed in Table IV of the Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445),<sup>4</sup> source of compressed air, constant-temperature oil bath accurately maintained at 120 C. (248 F.), small oil bath, 5-ml. pipette, 1-oz. wide-mouth bottle, and stop watch or timer.

(2) *REAGENT.*—The ortho-dichlorobenzene used for preparing the 2 per cent solution shall be the grade containing 96 to 99 per cent of ortho-dichlorobenzene and 1 to 4 per cent of para-dichlorobenzene and conforming to the following requirements:

	Limit	A.S.T.M. Method
Specific gravity, 20/4 C. {max..... 1.311 min..... 1.305}		D 268 <sup>a</sup>
Distillation range (first drop to dry point)..... {179 to 180.7 C. 354.2 to 357.3 F. }		D 86 <sup>b</sup>
Freezing point {max.—17.2 C. (+1 F.) min.—18.6 C. (-1.5 F.) }		"

<sup>a</sup> Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268).<sup>4a</sup>

<sup>b</sup> Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86).<sup>4</sup>

Freezing point shall be determined as follows: Use a thick walled test tube having a side tube and fitted into a slightly wider jacket.<sup>5</sup> Place the tube and jacket in a covered jar filled with a freezing mixture. Pour a sample of the ortho-dichlorobenzene in the inner test tube and close the test tube with a cork stopper containing a stirrer and a thermometer. The thermometer shall have subdivisions and an accuracy of 0.1 C. in the approximate range -25 to -15 C. and should be calibrated by comparison with a standard thermometer. Regulate the temperature of the freezing mixture to not more than 5 C. below the expected freezing point of the sample. Remove the test tube from its jacket and immerse it in the freezing mixture until the sample begins to freeze. Replace the test tube in its jacket and stir the sample vigorously. As the sample solidifies, the temperature will rise until the true freezing point is reached and then it will remain constant. Record this temperature as the freezing point of the sample.

(3) *PREPARATION OF 2 PER CENT SOLUTION.*—Weigh 0.2663 g. of the sam-

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

<sup>4a</sup> 1946 Book of A.S.T.M. Standards, Part II.

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>5</sup> The Beckman apparatus, described in *Physik. Chem.*, Part II, p. 683 (1888), has been found satisfactory.

ple into the wide-mouth bottle and add 10 ml. of ortho-dichlorobenzene to make a 2 per cent solution of polymer. Heat in the small oil bath with constant stirring to about 165 C. (329 F.) until all of the test sample has dissolved. If too much heat is applied, the test sample will decompose, which is indicated by a dark coloration, and the viscosity obtained will be low.

(4) PROCEDURE.—Clean and dry the viscosimeter, and suspend it in the constant-temperature bath for a sufficient time to reach bath temperature. Preheat the pipette and then transfer 5 ml. of the hot 2 per cent solution to the viscosimeter, using a piece of cotton over the tip of the pipette for a filter. Force the solution up the capillary above the upper mark, using compressed air, and then allow it to drain down through the capillary. Again force the solution up the capillary just above the upper mark using compressed air, allow it to drain down, and measure the time interval for the meniscus to pass from the upper to the lower mark, using the stop watch or timer. Repeat this operation three times and average the three measurements of efflux time. The three measurements should agree within 1 sec. Since the solution solidifies upon cooling, the viscosimeter should be removed from the bath and boiling ortho-dichlorobenzene immediately forced through the capillary to remove all of the solution.

Two check tests shall be conducted on each 2 per cent solution in two separate viscosimeters, and the results should agree within 0.01 centipoises.

(5) CALCULATIONS.—The kinematic viscosity of the 2 per cent solution shall be calculated in accordance with the provisions of Tentative Method D 445,<sup>4</sup> Appendix, Section A7, and the results in centistokes shall be converted to centipoises as follows:

$$\text{Viscosity in centipoises} = s \times V$$

where:

$s$  = specific gravity of 2 per cent solution, and

$V$  = kinematic viscosity of 2 per cent solution in centistokes.

(d) *Flow Temperature*.—Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (A.S.T.M. Designation: D 569).<sup>3</sup>

(e) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(f) *Tensile Strength*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638),<sup>3</sup> using a speed of testing of 0.25 in. per min.

(g) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using a fiber stress of 264 psi.

(h) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion.

(i) *Dielectric Strength*.—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> using test specimens  $\frac{1}{8}$  in. in thickness and immersed in oil.

(j) *Flammability*.—Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568).<sup>3</sup>

(k) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

(1) *Weight Loss on Heating.*—Test specimens 3 by 1 by  $\frac{1}{8}$  in. shall be conditioned for 48 hr. over anhydrous calcium chloride at  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall be weighed and then placed in a circulation air oven for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.). The specimens shall be supported flatwise on a screen in the oven. Upon removal from the oven, the specimens shall be cooled in a desiccator over anhydrous calcium chloride to  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall then be weighed and the percentage weight loss on heating calculated on the basis of the conditioned weight.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding material may be rejected at the option of the purchaser. Any rejections based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing.*—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking.*—Shipping containers shall be marked with the name of the material, form, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.



# Tentative Specifications for

## NONRIGID VINYL CHLORIDE-ACETATE RESIN PLASTICS<sup>1</sup>



**A.S.T.M. Designation: D 742-46 T**

ISSUED, 1943; REVISED, 1944, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover transparent, translucent, and opaque non-rigid plastics consisting of vinyl chloride-acetate resins of approximately 95 per cent vinyl chloride content and the necessary plasticizers, lubricants, stabilizers, dyes, pigments, and fillers. These specifications do not cover special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the types and grades of nonrigid plastics covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Types and Grades

2. These specifications cover three types and ten grades of nonrigid vinyl chloride-acetate resin plastics, the grades being classified in accordance with their hardnesses at 25 C. (77 F.), as shown in Table I.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

A low-temperature classification of these materials is also shown in Table I.

TABLE I.—TYPES AND GRADES OF NONRIGID VINYL CHLORIDE-ACETATE RESIN PLASTICS.

NOTE.—The hardness values and low-temperature classifications in this table are included in these specifications for purposes of classification only and are not specific requirements to be met.

Type	Grade	Durometer Hardness <sup>a</sup>	A.S.T.M. Hardness <sup>b</sup>	Low-Temperature Classification <sup>c</sup>
I. Unfilled (Plasticized with dioctyl phthalate or equivalent)	1...	90 ± 10	To be added at a later date	N
	2...	90 ± 10		N <sub>d</sub>
	3...			
	4...	80 ± 10		N
	5...	70 ± 10		F
	6...	60 ± 10		F
II. Cold resistant	7...	70 ± 10		F
	8...	70 ± 10		FF
III. Filled (plasticized with dioctyl phthalate or equivalent and containing an inert filler)	9...	70 ± 10		d
	10...	60 ± 10		d

<sup>a</sup> Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

<sup>b</sup> Standard Method of Test for Hardness of Rubber (A.S.T.M. Designation: D 314).<sup>3</sup>

<sup>c</sup> A low-temperature classification of FF indicates that the material does not fail at -70 F., a classification of F indicates that the material does not fail at -40 F., and a classification of N indicates that the material fails at -40 F. when tested in accordance with the Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736).<sup>3</sup>

<sup>d</sup> To be added at a later date.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE.—The physical properties of these non-rigid vinyl chloride-acetate resin plastics are chiefly dependent upon the kind and quantity of plasticizers and fillers used. A series of compounds with widely differing properties can be produced. The selection of types and grades for these specifications is arbitrary, but those shown in Table I represent the general purpose compositions.

### General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The color, transparency or opacity, form, and size shall be as specified by the purchaser in the contract or order.

NOTE.—Nonrigid vinyl chloride-acetate resin plastics are available in the following forms:

In a granulated or diced form suitable for feeding to extrusion or injection molding machines.

In rolls of tape  $3\frac{3}{4} \pm \frac{1}{4}$  in. in width and  $0.035 \pm 0.005$  in. in thickness suitable for feeding to extrusion machines.

In the form of sheeting supplied in rolls 10 to  $13\frac{1}{2}$  in. in diameter, wound on hard paper cores. Thicknesses and widths of sheeting are available as follows:

Thickness, in.	Width, in.
0.004.....	48
0.008.....	48
0.020.....	36
0.035.....	36
0.040.....	36

(c) The average thickness of the sheeting shall be within  $\pm 10$  per cent of the specified thickness.

(d) The nonrigid vinyl chloride-acetate resin plastic tape and sheeting covered by these specifications shall have a calendered surface.

(e) *Chemical Resistance.* — The resistance of the material to specific chemicals shall be as specified by the purchaser in the contract or order. In general, the effect of chemicals should be determined in accordance with the Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M. Designation: D 543),<sup>3</sup> using the test for stiffness, tensile strength, or

any other physical property included in these specifications to evaluate the effects.

### Detail Requirements

4. Test specimens, cut from sheet material molded under conditions specified by the manufacturer, shall conform to the requirements as to physical and electrical properties prescribed in Table II.

### Sampling

5. The material shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392)<sup>3</sup> where applicable; otherwise a sufficient sample shall be taken from each batch of material to be tested to establish conformance with these specifications. A batch shall be considered as a unit of manufacture as prepared for shipment, and may consist of a blend of two or more "production runs" of material. Unless otherwise specified, tests need not be run on more than one batch in every 1500 lb. of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Test Specimens.*—Test specimens shall be cut from sheets  $0.075 \pm 0.010$  in. in thickness, molded by compression under conditions specified by the manufacturer. Specimens shall be of the shape and dimensions specified in the individual test methods.

(b) *Conditioning Test Specimens.*—For those tests where conditioning is required, the test specimens of nonrigid vinyl chloride-acetate resin plastics shall

be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup> For tests in other than the Standard Laboratory Atmosphere, reference likewise shall be made to Methods D 618.

(e) *Tensile Strength and Elongation at Rupture*.—Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412),<sup>3</sup> using die C.

(f) *Stiffness*.—Tentative Method of Test for Stiffness in Flexure of Non-rigid Plastics (A.S.T.M. Designation: D 747).<sup>3</sup>

TABLE II.—DETAIL REQUIREMENTS FOR NONRIGID VINYL CHLORIDE-ACETATE RESIN PLASTICS.

	Type I						Type II		Type III	
	Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6	Grade 7	Grade 8	Grade 9	Grade 10
Specific gravity, (max. .... 25/25 C. (77/77 F.)) min. ....	1.29	1.26		1.24	1.22	1.19	1.22	1.21	1.45	1.40
Tensile strength, min., psi. ....	1.24	1.22		1.19	1.17	1.15	1.17	1.15	1.41	1.36
Elongation at rupture, min., per cent. ....	3 000	2 600		1 800	1 400	1 000	1 400	1 400	1 000	1 000
at 25 C. (max. ....)	250	300		330	360	360	330	330	250	250
at (-77 F.) min. ....	5 500	2 300		1 000	600	400	750	800	900	600
Stiffness, max., psi. ....	2 300	1 000		600	400	250	300	350	700	450
at -40 C. (-40 F.) ....	500 000	350 000		250 000	170 000	60 000	130 000	40 000	130 000	90 000
at -57 C. (-70 F.) ....	550 000	500 000		450 000	400 000	300 000	300 000	100 000	400 000	360 000
Tear resistance, min., lb. per in. ....	500	400		300	200	100	200	170	190	170
Brittle temperature, deg. Cent. ....	18	-22		-28	-33	-38	-54	-62	-29	-29
max., deg. Fahr. ....	0	-8		-18	-27	-36	-65	-80	-20	-20
Water absorption, max., per cent. ....										
plus soluble matter loss. ....	0.30	0.40		0.50	0.60	0.70	0.60	1.00	0.50	0.90
soluble matter loss. ....	0.10	0.12		0.15	0.18	0.20	0.25	0.25	0.10	0.20
Flammability (burning rate), max., sq. in. per sec. ....	0.020 in. thick	0.4		0.5	0.6	0.7	0.6	0.6	0.35	0.35
	0.040 in. thick	a		a	0.1	0.2	0.3	0.3	0.15	0.25
Weight loss on heating (72 hr. at 82 C. (180 F.)) max., per cent. ....	1.5	1.7		1.9	2.2	2.4	2.4	2.8	0.55	0.65
Increase in stiffness at 25 C. (77 F.) after heating, max., per cent. ....	30	30		30	30	30	35	40	30	30
Increase in brittle temperature after heating, max., deg. Cent. ....	2	2		2	2	2	6	7	3	3
deg. Fahr. ....	4	4		4	4	4	10	12	5	5
Thermal stability. ....	2	2		2	2	2	2	2	2	2

<sup>a</sup> Self-extinguishing.

(c) *Test Conditions*.—Tests shall be conducted in a standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(d) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(g) *Tear Resistance*.—Standard Method of Test for Tear Resistance of Vulcanized Rubber (A.S.T.M. Designation: D 624),<sup>3</sup> using die B.

(h) *Brittle Temperature*.—Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746).<sup>3</sup>

(i) *Water Absorption*.—Standard Method of Test for Water Absorption

of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using test specimens 0.075 in. in thickness and the 24-hr. immersion procedure. The percentage of water absorbed which is gain in weight plus soluble matter lost, and the percentage of soluble matter as determined by this method shall be reported.

(j) *Flammability*.—Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568),<sup>3</sup> using test specimens 0.020 and 0.040 in. in thickness.

(k) *Weight Loss on Heating*.—Test specimens 1 by 5 by 0.075 in. shall be heated for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.) in a forced circulation air oven. The specimens shall be placed on a smooth, horizontal, chromium-plated surface and shall be turned over after 48 hr. of exposure. Specimens shall be conditioned for 48 hr. in air at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity before weighing both before and after heating. The loss of weight shall be calculated as a percentage of the original weight.

(l) *Stiffness After Heating*.—Stiffness in flexure at 25 C. (77 F.) after heating shall be determined in accordance with the method referred to in Paragraph (f), using specimens cut from those heated and reconditioned as described in Paragraph (k). The increase in stiffness at 25 C. (77 F.) after heating shall be calculated as a percentage of the original value obtained for the stiffness in flexure as determined in accordance with Paragraph (f).

(m) *Brittle Temperature After Heating*.—Brittle temperature after heating shall be determined in accordance with the method referred to in Paragraph (h), using specimens cut from those heated and reconditioned as described in Paragraph (k). The increase in brittle temperature after heating shall be cal-

culated as the difference in degrees Centigrade or Fahrenheit between the brittle temperatures before and after heating.

(n) *Thermal Stability*.—Tentative Method of Test for Short-Time Stability at Elevated Temperatures of Plastics Containing Chlorine (A.S.T.M. Designation: D 793).<sup>3</sup>

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

**Packing and Marking**

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*. — Shipping containers shall be marked with the name of the material, type, grade, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# Tentative Specifications for NONRIGID ETHYL CELLULOSE PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 743-44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover nonrigid plastics consisting of ethyl cellulose and the necessary plasticizers, dyes, pigments, fillers, and other ingredients, compounded to produce a thermoplastic composition suitable for injection, compression, or extrusion molding. These specifications do not cover special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the types and grades of nonrigid plastics covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types and Grades

2. These specifications cover two types and six grades of nonrigid ethyl cellulose

plastics as shown in Table I. The hardness values at 25 C. (77 F.) and a low-temperature classification of these materials are also shown in Table I.

TABLE I.—TYPES AND GRADES OF NONRIGID  
ETHYL CELLULOSE PLASTICS.

NOTE.—The hardness values and low-temperature classifications in this table are included in these specifications for purposes of classification only and are not specific requirements to be met.

Type	Grade	Durometer Hardness <sup>a</sup>	A.S.T.M. Hardness <sup>b</sup>	Low-Temperature Classification <sup>c</sup>
I. Molding.....	1...	80 ± 10	46 ± 22	N
	2...	70 ± 10	68 ± 22	F
	3...	90 ± 10	23 ± 22	FF
II. Extrusion....	4...	70 ± 10	68 ± 22	F
	5...	70 ± 10	68 ± 22	N
	6...	80 ± 10	46 ± 22	F

<sup>a</sup> Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

<sup>b</sup> Standard Method of Test for Hardness of Rubber (A.S.T.M. Designation: D 314).<sup>3</sup>

<sup>c</sup> A low-temperature classification of FF indicates that the material does not fail at -70 F., a classification of F indicates that the material does not fail at -40 F., and a classification of N indicates that the material fails at -40 F. when tested in accordance with the Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736).<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE.—The physical properties of these non-rigid ethyl cellulose plastics are chiefly dependent upon the kind and quantity of plasticizers and fillers used. A series of compounds with small differences in properties can be produced. The selection of types and grades for these specifications is arbitrary.

## General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

mined in accordance with the Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M. Designation: D 543),<sup>3</sup> using the test for stiffness, tensile strength, or any other physical property included in these specifications to evaluate the effects.

### Detail Requirements

4. Test specimens, either cut from sheet material or molded under conditions specified by the manufacturer.

TABLE II.—DETAIL REQUIREMENTS FOR NONRIGID ETHYL CELLULOSE PLASTICS.

	Type I			Type II		
	Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6
Specific gravity, 25/25 C. (77/77 F.), max. ....	1.14	1.20	1.20	1.14	1.20	1.20
Tensile strength, min., psi. ....	1 400	400	1 800	600	200	800
Elongation at rupture, min., per cent. ....	80	70	70	60	30	60
(at 25 C. (+77 F.) .....	2 000	2 500	2 500	1 500	2 000	2 000
Stiffness, max., psi. ....	75 000	50 000	90 000	70 000	50 000	65 000
(at -40 C. (-40 F.) .....	...	...	...	...	...	...
(at -57 C. (-70 F.) .....	180	140	140	80	70	100
Tear resistance, min., lb. per in. ....	-25	-20	-57	-25	-15	-40
Brittle temperature, max. ....	-13	-4	-70	-13	+5	-40
(deg. Cent.) .....	...	...	...	...	...	...
(deg. Fahr.) .....	1.5	1.5	2.8	1.7	3.0	2.8
Water absorption, max., (weight gain plus soluble matter loss. ....	0.2	0.2	0.3	0.2	0.2	0.3
per cent (soluble matter loss. ....	20	25	20	20	40	25
Deformation under load of 100 (at 50 C. (122 F.) .....	40	45	40	40	80	50
psi., max., per cent (at 70 C. (158 F.) .....	0.7	0.7	1.0	0.7	0.7	1.0
Flammability (burning rate), max., sq. in. per sec. (specimen 0.040 in. thick) .....	2.0	2.0	2.5	2.5	4.5	2.5
Weight loss on heating (72 hr. at 82 C. (180 F.)) max., per cent .....	50	40	40	35	25	40
Increase in stiffness at 25 C. (77 F.) after heating, max., per cent. ....	8	8	6	8	8	6
Increase in brittle temperature after heating, max. ....	15	15	10	15	15	10
(deg. Cent.) .....	1 050	300	...	450	150	...
(deg. Fahr.) .....	...	...	...	...	...	...
Tensile strength after heating, min., psi. ....	...	...	...	...	...	...
Thermal stability .....	All samples shall be homogeneously soluble and shall have a viscosity of at least 18 centipoises.					
Dielectric strength (short-time test), min., v. per mil. ....						
	To be specified at a later date.					

(b) The color, transparency or opacity, form, and particle size shall be as specified by the purchaser in the contract or order.

NOTE.—Nonrigid ethyl cellulose plastics are available in the following forms:

Type I. Molding: granular sizes of  $\frac{1}{8}$  to  $\frac{1}{2}$  in.

Type II. Extrusion: granular sizes of  $\frac{1}{8}$  to  $\frac{1}{2}$  in., or cut slabs from mixing rolls.

(c) *Chemical Resistance.*—The resistance of the material to specific chemicals shall be as specified by the purchaser in the contract or order. In general, the effect of chemicals should be deter-

shall conform to the requirements as to physical and electrical properties prescribed in Table II.

## Sampling

5. The material shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392)<sup>3</sup> where applicable; otherwise a sufficient sample shall be taken from each batch of mate-



rial to be tested to establish conformance with these specifications. A batch shall be considered as a unit of manufacture as prepared for shipment, and may consist of a blend of two or more "production runs" of material.

#### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Test Specimens*.—Test specimens shall be cut from sheets compression molded from the material, or shall be molded by compression under conditions specified by the manufacturer. Sheets so tested shall be  $0.075 \pm 0.010$  in. in thickness, unless otherwise specified, and test specimens shall be of the shape and dimensions specified in the individual test methods.

(b) *Conditioning Test Specimens*.—For those tests where conditioning is required, the test specimens of nonrigid ethyl cellulose plastics shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup> For tests in other than the Standard Laboratory Atmosphere, reference likewise shall be made to Methods D 618.

(c) *Test Conditions*.—Tests shall be conducted in a standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(d) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Tensile Strength and Elongation at Rupture*.—Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412),<sup>3</sup> using die C.

(f) *Stiffness*.—Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (A.S.T.M. Designation: D 747).<sup>3</sup>

(g) *Tear Resistance*.—Standard Method of Test for Tear Resistance of Vulcanized Rubber (A.S.T.M. Designation: D 624),<sup>3</sup> using die B.

(h) *Brittle Temperature*.—Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746).<sup>3</sup>

(i) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using test specimens 1 by 3 by 0.075 in. and the 24-hr. immersion procedure. The percentage of water absorbed which is gain in weight plus soluble matter lost and the percentage of soluble matter as determined by this method shall be reported.

(j) *Deformation Under Load*.—Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621),<sup>3</sup> loading unconditioned specimens at 100 psi. for 3 hr. at the required temperature.

(k) *Flammability*.—Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568),<sup>3</sup> using only test specimens 0.040 in. in thickness.

(l) *Weight Loss on Heating*.—Test specimens 1 by 5 by 0.075 in. shall be heated for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.) in a forced circulation air oven. The specimens shall be placed on a smooth, horizontal, chromium-plated surface and shall be turned over after 48 hr. of exposure. Specimens shall be conditioned for 48 hr. in air at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity before weighing both before and after heating. The loss of weight shall be calculated as a percentage of the original weight.

(m) *Stiffness After Heating*.—Stiffness

in flexure at 25 C. (77 F.) after heating shall be determined in accordance with the method referred to in Paragraph (f), using specimens cut from those heated and reconditioned as described in Paragraph (l). The increase in stiffness at 25 C. (77 F.) after heating shall be calculated as a percentage of the original value obtained for the stiffness in flexure as determined in accordance with Paragraph (f).

(n) *Brittle Temperature After Heating.*—Brittle temperature after heating shall be determined in accordance with the method referred to in Paragraph (h), using specimens cut from those heated and reconditioned as described in Paragraph (l). The increase in brittle temperature after heating shall be calculated as the difference in degrees Centigrade or Fahrenheit between the brittle temperatures before and after heating.

(o) *Tensile Strength After Heating.*—Tensile strength after heating shall be determined in accordance with the method referred to in Paragraph (e), using specimens cut from those heated and reconditioned as described in Paragraph (l).

(p) *Thermal Stability.*—After initial conditioning and heating the material for 72 hr. in accordance with the method described in Paragraph (l), a test specimen weighing  $7.5 \pm 0.1$  g. shall be brought to room temperature in a desiccator filled with  $\text{CaCl}_2$ . The specimen shall be placed in a wide-mouth 8-oz. bottle, and dissolved in 92.5 g. of a solvent of 80 parts by weight of toluol and 20 parts by weight of 95 per cent ethanol. After being tumbled overnight in a shaker at a speed of 6 to 10 rpm., the solution shall be examined for homogeneity. If noncolloided portions do not appear, the solution shall be brought to thermal equilibrium by holding it in a water bath at the test temperature for at least 1 hr. The viscosity of the

solution in centipoises at 25 C. (77 F.) shall be determined by any acceptable method. The following procedures are recommended:

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88),<sup>3a</sup> except that the efflux time in seconds shall be converted to centipoises by the following formula:

Viscosity in centipoises =

$$100 \times s \times \left[ (0.0022 \times t) - \frac{1.8}{t} \right]$$

where:

$s$  = specific gravity of the solution, and  
 $t$  = efflux time in seconds.

Tentative Method of Test for Kinematic Viscosity (A.S.T.M. Designation: D 445),<sup>3a</sup> using Method B and the series 300 modified Ostwald viscosimeter, except that the result in centistokes shall be converted to centipoises by the following formula:

$$\text{Viscosity in centipoises} = s \times V$$

where:

$s$  = specific gravity of the solution, and  
 $V$  = kinematic viscosity in centistokes.

NOTE.—Since the strength and toughness of these plastics depends upon having an ethyl cellulose of sufficiently high molecular weight, it is advisable to carry out this test to ensure that degraded material does not develop upon use.

(q) *Dielectric Strength.*—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> using the short-time test in oil with a test specimen 6 by 6 by 0.075 in.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for

<sup>3a</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

testing each batch. The average result for the specimens tested shall conform with the limits prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails,

the batch of molding material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers so constructed as to insure acceptance by common or other carriers, for safe transportation, at the lowest rate, to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order

# Tentative Specifications for

## NONRIGID VINYL CHLORIDE PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 744 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover nonrigid plastics consisting of vinyl chloride resins and the necessary plasticizers, stabilizers, lubricants, dyes, pigments, and fillers. These specifications do not cover special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the types and grades of nonrigid plastics covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Types and Grades

2. These specifications cover four types and nine grades of nonrigid vinyl chloride plastics as shown in Table I, the grades being classified in accordance with their hardnesses at 25 C. (77

F.). A low-temperature classification of these materials is also shown in Table I.

TABLE I.—TYPES AND GRADES OF NONRIGID VINYL CHLORIDE PLASTICS.

NOTE.—The hardness values and low-temperature classifications in this table are included in these specifications for purposes of classification only and are not specific requirements to be met.

Type	Grade	Durometer Hardness <sup>a</sup>	A.S.T.M. Hardness <sup>b</sup>	Low-Temperature Classification <sup>c</sup>
I. Unfilled (plasticized with dioctyl phthalate or equivalent)	1...	90 ± 10	23 ± 4	N
	2...	80 ± 10	30 ± 5	N
	3...	80 ± 10	38 ± 5	F
	4...	70 ± 10	48 ± 5	F
II. Heat and flame resistant	5...	80 ± 10	40 ± 7	N
III. Cold resistant	6...	80 ± 10	25 ± 6	F
	7...	80 ± 10	30 ± 7	FF
IV. Filled	8...	90 ± 10	22 ± 5	N
	9...	90 ± 10	27 ± 6	N

<sup>a</sup> Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

<sup>b</sup> Standard Method of Test for Hardness of Rubber (A.S.T.M. Designation: D 314).<sup>3</sup>

<sup>c</sup> A low-temperature classification of FF indicates that the material does not fail at -70 F., a classification of F indicates that the material does not fail at -40 F., and a classification of N indicates that the material fails at -40 F. when tested in accordance with the Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736).<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

NOTE.—The physical properties of these non-rigid vinyl chloride plastics are chiefly dependent upon the kind and quantity of plasticizers and fillers used. A series of compounds with small differences in properties can be produced. The selection of types and grades for these specifications is arbitrary.

### General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(c) *Chemical Resistance.*—The resistance of the material to specific chemicals shall be as specified by the purchaser in the contract or order. In general, the effect of chemicals should be determined in accordance with the Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M. Designation: D 543),<sup>3</sup> using the test for stiffness, tensile strength, or any other

TABLE II.—DETAIL REQUIREMENTS FOR NONRIGID VINYL CHLORIDE PLASTICS.

	Type I				Type II	Type III		Type IV	
	Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6	Grade 7	Grade 8	Grade 9
Specific gravity, 25/25 C. (77/77 F.) {max. .... {min. ....	1.38 1.22	1.34 1.21	1.31 1.19	1.30 1.18	1.40 1.29	1.39 1.21	1.38 1.19	1.52 1.39	1.65 1.49
Tensile strength, min., psi. ....	2 600	2 200	1 800	1 400	2 200	2 600	2 200	1 400	1 000
Elongation at rupture, min., per cent. ....	300	330	360	390	250	330	360	200	250
{at 25 C. (+77 F.) {max. .... {min. ....	2 750 1 800	1 650 900	1 200 700	800 500	1 100 700	2 200 1 000	1 500 800	b b	b b
Stiffness, psi. {at -40 C. (-40 F.), max. .... {at -57 C. (-70 F.), max. ....	450 000 600 000	350 000 500 000	275 000 450 000	200 000 400 000	700 000 1 000 000	170 000 300 000	100 000 130 000	b b	b b
Tear resistance, min., lb. per in. ....	550	450	350	250	400	400	300	350	250
Brittle temperature, deg. Cent. ....	-20	-25	-29	-33	-2	-46	-51	b	b
deg. Fahr. ....	-4	-13	-20	-27	+28	-50	-60	b	b
Water absorption, {weight gain plus {soluble matter loss. ....	0.40 0.10	0.50 0.15	0.65 0.15	0.60 0.20	0.60 0.20	1.75 1.50	2.25 2.00	b a	b b
Flammability (burning rate), max., {0.020 in. thick. .... {0.040 in. thick. ....	0.4 a	0.4 a	0.4 a	0.5 0.2	a a	0.6 0.3	0.6 0.3	b a	b b
Weight loss on heating (72 hr. at 82 C. (180 F.)), max., per cent. ....	0.6	0.7	0.8	0.8	0.8	2.0	2.5	0.5	1.7
Increase in stiffness at 25 C. (77 F.) after heating, max., per cent. ....	15	15	15	15	15	30	30	15	30
Increase in brittle temperature after {deg. Cent. .... {deg. Fahr. ....	2 4	2 4	2 4	2 4	2 4	5 9	5 9	b b	b b
Thermal stability, max., mg. HCl per g. Dielectric strength (short-time test), min., v. per mil. ....	425	400	400	350	350	350	325	400	350
Oil resistance, max., per cent loss in weight. ....	.....	.....	.....	.....	0.15	.....	.....	.....	.....
Deformation under load, 100 psi. pressure at 70 C. (158 F.): Total deformation, max., per cent. ....	25	30	35	45	35	25	35	15	30
Recovery, min., per cent. ....	75	75	65	60	65	60	70	70	75

<sup>a</sup> Self-extinguishing.

<sup>b</sup> To be specified at a later date.

(b) The color, transparency or opacity, shape, and size shall be as specified by the purchaser in the contract or order.

NOTE.—Nonrigid vinyl chloride plastics are available in the following forms:

In a diced or granulated form suitable for feeding to extrusion or injection molding machines.

In the form of tape and sheeting supplied in rolls 10 to 18 in. in diameter, wound on hard paper cores. Material  $3\frac{1}{4}$  to 33 in. in width is available in any thickness from 0.010 to  $0.030 \pm 0.002$  in.

physical property included in these specifications to evaluate the effects.

### Detail Requirements

4. Test specimens, either cut from sheet material or molded under conditions specified by the manufacturer, shall conform to the requirements as to physical and electrical properties prescribed in Table II.

### Sampling

5. The material shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392)<sup>3</sup> where applicable; otherwise a sufficient sample shall be taken from each batch of material to be tested to establish conformance with these specifications. A batch shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material. Unless otherwise specified, tests need not be run on more than one batch in every 1500 lb. of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Test Specimens*.—Test specimens for tests other than hardness and flammability shall be cut from sheets  $0.075 \pm 0.010$  in. in thickness, molded by compression under conditions specified by the manufacturer. Specimens shall be of the shape and dimensions specified in the individual test methods.

(b) *Conditioning Test Specimens*.—Molded test specimens of nonrigid vinyl chloride plastics are not sensitive to humidity, but shall be conditioned for at least 48 hr. in air at  $25 \pm 1$  C. ( $77 \pm 2$  F.) immediately prior to testing. Samples for tests at subatmospheric temperatures shall be exposed an additional 24 hr. to air at the test temperatures before testing, unless otherwise specified in the testing methods or in these specifications. For test at temperatures above 25 C. (77 F.), the secondary conditioning period shall be in air at the test temperatures, and shall

be long enough to insure thermal equilibrium but in no case less than 2 hr.

(c) *Test Conditions*.—Tests shall be conducted in a standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(d) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Tensile Strength and Elongation at Rupture*.—Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412),<sup>3</sup> using die C.

(f) *Stiffness*.—Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (A.S.T.M. Designation: D 747).<sup>3</sup>

(g) *Tear Resistance*.—Standard Method of Test for Tear Resistance of Vulcanized Rubber (A.S.T.M. Designation: D 624),<sup>3</sup> using die B.

(h) *Brittle Temperature*.—Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746).<sup>3</sup>

(i) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using test specimens 0.075 in. in thickness and the 24-hr. immersion procedure. The percentage of water absorbed which is gain in weight plus soluble matter lost, and the percentage of soluble matter as determined by this method shall be reported.

(j) *Flammability*.—Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568),<sup>3</sup> using test specimens both 0.020 and 0.040 in. in thickness.

(k) *Weight Loss on Heating*.—Test specimens 1 by 5 by 0.075 in. shall be heated for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.) in a forced circulation air oven. The

specimens shall be placed on a smooth, horizontal, chromium-plated surface and shall be turned over after 48 hr. of exposure. Specimens shall be conditioned for 48 hr. in air at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity before weighing both before and after heating. The loss of weight shall be calculated as a percentage of the original weight.

(l) *Stiffness After Heating*.—Stiffness in flexure at 25 C. (77 F.) shall be determined in accordance with the method referred to in Paragraph (f), using specimens cut from those heated and reconditioned as described in Paragraph (k). The increase in stiffness at 25 C. (77 F.) after heating shall be calculated as a percentage of the original value obtained for the stiffness in flexure test as determined in accordance with Paragraph (f).

(m) *Brittle Temperature After Heating*.—Brittle temperature shall be determined in accordance with the method referred to in Paragraph (h), using specimens cut from those heated and reconditioned as described in Paragraph (k). The increase in brittle temperature after heating shall be calculated as the difference in degrees Centigrade or Fahrenheit between the brittle temperatures before and after heating.

(n) *Thermal Stability*.—Tentative Method of Test for Short-Time Stability at Elevated Temperatures of Plastics Containing Chlorine (A.S.T.M. Designation: D 793).<sup>3</sup>

(o) *Dielectric Strength*.—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149).<sup>3</sup> using the short-time test in oil with a test specimen 6 by 6 by 0.075 in.

(p) *Oil Resistance*.—Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M.

Designation: D 543).<sup>3</sup> using specimens 0.075 in. in thickness. The oil shall have an aniline point of  $70 \pm 2$  C. when determined by the Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611).<sup>4</sup> and a Saybolt Universal viscosity of  $150 \pm 10$  sec. at 100 F. when determined by the Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>4</sup>

(q) *Deformation Under Load*.—Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621).<sup>3</sup> loading unconditioned specimens at 100 psi. at 70 C. (158 F.).

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of material may be rejected at

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.



the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications, shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### **Packing and Marking**

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure ac-

ceptance by common or other carriers for safe transportation, at the lowest rate, to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# Tentative Specifications for NONRIGID VINYL BUTYRAL PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 745 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover nonrigid plastics consisting of vinyl butyral resins, obtained by the partial acetalization with butyraldehyde of the product obtained in the complete or partial hydrolysis of vinyl acetate resins, and the necessary plasticizers, dyes, pigments, and modifying agents. These specifications do not cover special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the types and grades of nonrigid plastics covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types and Grades

2. These specifications cover two types and eight grades of nonrigid vinyl butyral plastics as shown in Table I,

the grades being classified in accordance with their hardnesses at 25 C. (77 F.).

TABLE I.—TYPES AND GRADES OF NONRIGID VINYL BUTYRAL PLASTICS.

NOTE.—The hardness values and low-temperature classifications in this table are included in these specifications for purposes of classification only and are not specific requirements to be met.

Type	Grade	Durometer Hardness <sup>a</sup>	A.S.T.M. Hardness <sup>b</sup>	Low-Temperature Classification <sup>c</sup>
I. Thermoplastic	1..	60 ± 10	...	F
	2..	50 ± 10	...	F
	3..	50 ± 10	75 ± 25	F
	4..	40 ± 10	100 ± 40	F
II. Curing (contains "cross - linking" agents, which cause transformation from the thermoplastic to "thermoset" characteristics when the composition is properly cured)	5..	70 ± 10	35 ± 15	F
	6..	60 ± 10	50 ± 20	F
	7..	40 ± 10	100 ± 40	FF
	8..	20 ± 10	190 ± 50	FF

<sup>a</sup> Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

<sup>b</sup> Standard Method of Test for Hardness of Rubber (A.S.T.M. Designation: D 314).<sup>3</sup>

<sup>c</sup> A low-temperature classification of FF indicates that the material does not fail at -70 F., and a classification of F indicates that the material does not fail at -40 F. when tested in accordance with the Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736).<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

A low-temperature classification of these materials is also shown in Table I.

NOTE.—The physical properties of these nonrigid vinyl butyral plastics are chiefly dependent upon the kind and quantity of plasticizers and fillers used and the presence or absence of "cross-linking" agents. Compounds varying by small increments through a wide range of properties can be produced. The selection of types and grades for these specifications is arbitrary.

### General Requirements

3. (a) The material shall be of uniform composition and so compounded as

the contract or order. In general, the effect of chemicals should be determined in accordance with the Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M. Designation: D 543),<sup>3</sup> using the test for stiffness, tensile strength, or any other physical property included in these specifications to evaluate the effects.

### Detail Requirements

4. Test specimens, either cut from sheet material, which for type II shall be cured under conditions specified by

TABLE II.—DETAIL REQUIREMENTS OF NONRIGID VINYL BUTYRAL PLASTICS.

	Type I				Type II			
	Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6	Grade 7	Grade 8
Specific gravity, 25/25 C. (77/77 F.) {max. .... min. ....			1.50 1.05	1.50 1.05	1.50 1.05	1.50 1.05	1.50 1.05	1.30 1.05
Tensile strength, min., psi.....			1 400	1 000	1 800	1 400	1 000	1 000
Elongation at rupture, min., per cent.....			250	250	200	250	250	300
Stiffness, max., psi. {at 25 C. (77 F.)..... at -40 C. (-40 F.)..... at -57 C. (-70 F.).....			a 150 000 250 000	a 70 000 200 000	a 200 000 350 000	a 100 000 250 000	a 70 000 200 000	a 40 000 150 000
Tear resistance, min., lb. per in.....			200	a	200	200	75	40
Brittle temperature, max. {deg. Cent..... deg. Fahr.....			-40 -40	-45 -49	-35 -31	-40 -40	-35 -31	-55 -67
Water absorption, {weight gain plus soluble max., per cent {matter loss..... soluble matter loss.....			3.0 1.0	3.0 1.0	3.0 1.0	3.0 1.0	3.0 1.0	3.5 1.0
Flammability (burning rate), max., sq. in. per sec. (0.040 in. thick).....			0.5	0.5	0.5	0.5	0.5	0.5
Weight loss on heating (72 hr. at 82 C. (180 F.)), max., per cent.....			2	2	2	2	2	4
Increase in stiffness after {at 25 C. (77 F.)..... heating, max., per cent {at -40 C. (-40 F.).....			10	10	25	25	25	25
Decrease in tensile strength after heating, max., per cent.....			15	15	15	15	15	15
Decrease in elongation after heating, max., per cent.....			15	15	15	15	15	15
Increase in brittle temperature {deg. Cent..... after heating, max. {deg. Fahr.....			5 9	5 9	10 18	10 18	10 18	10 18

<sup>a</sup> To be specified at a later date.

to conform to the requirements prescribed in these specifications.

(b) The color, transparency or opacity, form and size shall be as specified by the purchaser in the contract or order.

NOTE.—Nonrigid vinyl butyral plastics are available in the following forms:

In milled or calendered slabs, sheets, and rolls.

In the form of compounded dough, and dopes and coating solutions.

(c) *Chemical Resistance*.—The resistance of the material to specific chemicals shall be as specified by the purchaser in

the manufacturer, or molded under conditions specified by the manufacturer, shall conform to the requirements as to physical properties prescribed in Table II.

### Sampling

5. The material shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392)<sup>3</sup> where

applicable; otherwise a sufficient sample shall be taken from each batch of the material to be tested to establish conformance with these specifications. A batch shall be considered as a unit of manufacture as prepared for shipment, and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Test Specimens*.—Test specimens shall be cut from sheets molded from the material, or shall be molded by compression under conditions specified by the manufacturer. Sheets so tested shall be  $0.075 \pm 0.010$  in. in thickness, unless otherwise specified, and test specimens shall be of the shape and dimensions specified in the individual testing methods.

(b) *Conditioning Test Specimens*.—For those tests where conditioning is required, the test specimens of nonrigid vinyl butyral plastics shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup> For tests in other than the Standard Laboratory Atmosphere, reference likewise shall be made to Methods D 618.

(c) *Test Conditions*.—Tests shall be conducted in a standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the testing methods or in these specifications.

(d) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Tensile Strength and Elongation at Rupture*.—Standard Methods of Ten-

sion Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412),<sup>3</sup> using die C.

(f) *Stiffness*.—Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (A.S.T.M. Designation: D 747).<sup>3</sup>

(g) *Tear Resistance*.—Standard Method of Test for Tear Resistance of Vulcanized Rubber (A.S.T.M. Designation: D 624),<sup>3</sup> using die B.

(h) *Brittle Temperature*.—Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746).<sup>3</sup>

(i) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using test specimens 1 by 3 by  $0.075 \pm 0.010$  in. and the 24-hr. immersion procedure. The percentage of water absorbed which is gain in weight plus soluble matter lost and the percentage of soluble matter as determined by this method shall be reported.

(j) *Flammability*.—Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: 568),<sup>3</sup> using test specimens 0.040 in. in thickness.

(k) *Weight Loss on Heating*.—Test specimens 1 by 5 by  $0.075$  in. shall be heated for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.) in a forced circulation air oven. The specimens shall be placed on a smooth, horizontal, chromium-plated surface and shall be turned over after 48 hr. of exposure. Specimens shall be conditioned for 48 hr. in air at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity before weighing both before and after heating. The loss of weight shall be calculated as a percentage of the original weight.

(l) *Stiffness After Heating*.—Stiffness in flexure at  $-40$  C. ( $-40$  F.) shall be determined in accordance with the method referred to in Paragraph (f), using specimens cut from those heated and reconditioned as described in Paragraph (k). The increase in stiffness at  $-40$  C.

(-40 F.) after heating shall be calculated as a percentage of the original value obtained for the stiffness in flexure test at -40 C. (-40 F.) as determined in accordance with Paragraph (f).

(m) *Brittle Temperature After Heating.*—Brittle temperature shall be determined in accordance with the method referred to in Paragraph (h), using specimens cut from those heated and reconditioned as described in Paragraph (k). The increase in brittle temperature after heating shall be calculated as the difference in degrees Centigrade or Fahrenheit between the brittle temperatures before and after heating.

(n) *Tensile Strength and Elongation at Rupture After Heating.*—The tensile strength and elongation at rupture shall be determined in accordance with the method referred to in Paragraph (e), using specimens cut from those heated and reconditioned as described in Paragraph (k). The decrease in tensile strength and in elongation at rupture after heating shall be calculated as a percentage of the original value obtained for tensile strength and elongation at rupture as determined in accordance with Paragraph (e).

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection

be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing.*—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking.*—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which the shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Tentative Specifications for*

## CELLULOSE ACETATE PLASTIC SHEETS<sup>1</sup>



A.S.T.M. Designation: D 786 - 46 T

ISSUED, 1944; REVISED, 1945, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These specifications cover rigid plastic sheets made from cellulose acetate and plasticizers, with or without the addition of dyes and up to 2.5 per cent of pigment color. These specifications do not cover special sheets compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the type of material covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

(b) Filled or heavily pigmented sheets are not covered by these specifications as their properties will vary depending upon the thickness of the sheets and the degree of pigmentation.

### Type

2. These specifications cover the following two types of cellulose acetate plastic sheet containing not more than 2.5 per cent inert pigment or filler:

*Type I*.—General purpose material.

*Type II*.—Aircraft material.

### General Requirements

3. (a) The material shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) Requirements for size, dimensional tolerances, surface finish, and color shall be as specified by the purchaser in the contract or order (Notes 1, 2, and 3).

NOTE 1.—Standard size trimmed sheets are  $20 \pm \frac{1}{16}$  in. by  $50 \pm \frac{1}{16}$  in. by nominal thickness from 0.005 to 0.250 in. The thickness may vary from nominal thickness at any point in the sheet within the limits shown in Table I.

NOTE 2.—Sheets may be finished on one or both sides with any of the following finishes:

- a.—Polished,
- b.—Matte, and
- c.—Special embossed finishes such as linen, leather, etc.

NOTE 3.—Type I and type II sheets may be clear or colored transparent. Type I sheets may also be translucent.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by the Administrative Committee on Standards, September 9, 1946.

### Detail Requirements

4. The material shall conform to the requirements as to physical and electrical properties prescribed in Table II.

### Sampling

5. A sample shall be selected at random from each lot of material sufficient to determine the conformance of the material to these specifications. If the sheets are of such thickness that the necessary test specimens cannot be ob-

TABLE I.—THICKNESS OF SHEETS.

Nominal Thickness, in.	Actual Thickness, in.	
	Maximum	Minimum
0.005.....	0.006	0.0045
0.010.....	0.011	0.009
0.015.....	0.017	0.014
0.020.....	0.022	0.018
0.025.....	0.027	0.023
0.030.....	0.033	0.028
0.040.....	0.044	0.037
0.050.....	0.054	0.047
0.060.....	0.064	0.057
0.070.....	0.074	0.067
0.080.....	0.084	0.076
0.090.....	0.095	0.086
0.100.....	0.105	0.095
0.110.....	0.115	0.105
0.125.....	0.132	0.120
0.135.....	0.144	0.130
0.150.....	0.158	0.144
0.160.....	0.168	0.153
0.175.....	0.183	0.168
0.185.....	0.195	0.177
0.200.....	0.210	0.190
0.225.....	0.240	0.215
0.250.....	0.265	0.240

tained, specimens may be cut from sheets  $\frac{1}{8}$  in. in thickness and prepared from the same plastic composition under similar production conditions.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials.

#### (a) Conditioning Test Specimens.—

For those tests where conditioning is required, the test specimens of cellulose acetate plastic sheets shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of

TABLE II.—DETAIL REQUIREMENTS FOR CELLULOSE ACETATE PLASTIC SHEETS.

	Thickness, in.			
	0.005 to 0.030	0.031 to 0.060	0.061 to 0.100	0.101 to 0.250
TYPE I—GENERAL PURPOSE				
Specific gravity, 25/25 C. (77/77 F.) { min.....	1.27	1.27	1.27	1.27
max.....	1.34	1.34	1.34	1.34
Tensile strength, min., psi.....	5300	4700	4500	4000
Elongation, min., per cent.....	25	25	25	25
Impact strength (Izod), min., ft.-lb. per in. of (at +25 C. (77 F.) notch (at -40 C. (-40 F.)	...	...	1.4	1.4
Haze value (colorless), min., per cent.....	...	...	0.25	0.25
Water absorption, { Weight gain max., per cent { plus soluble matter loss..	9.0	7.5	7.0	4.5
Soluble matter loss.....	4.0	2.0	1.0	0.5
Heat distortion tem- perature at 264 psi. { deg. Cent... fiber stress, min. { deg. Fahr... Weight loss on heating, max., per cent.....	...	...	54	54
	...	...	131	131
Flammability (burning rate), max.: Thickness, in.: Over 0.050, in. per min..... 0.031 to 0.050, sq. in. per sec.. 0.016 to 0.030, sq. in. per sec.. 0.011 to 0.015, sq. in. per sec.. 0.005 to 0.010, sq. in. per sec..	...	6 0.4 0.5 1.0 1.5	4 ...	4 ...
To be specified at a later date				
TYPE II—AIRCRAFT				
Specific gravity, 25/25 C. (77/77 F.) { min.....	1.27	1.27	1.27	1.27
max.....	1.34	1.34	1.34	1.34
Tensile strength, min., psi.....	6000	5500	5000	4000
Elongation, min., per cent.....	25	25	25	25
Impact strength (Izod), min., ft.-lb. per in. of (at +25 C. (77 F.) notch (at -40 C. (-40 F.)	...	...	1.4	1.4
Haze value (colorless), max., per cent.....	2.0	3.0	5.0	8.0
Water absorption, { Weight gain max., per cent { plus soluble matter loss..	7.0	6.5	5.5	3.5
Soluble matter loss.....	4.0	2.0	1.0	0.5
Heat distortion tem- perature at 264 psi. { deg. Cent... fiber stress, min. { deg. Fahr... Weight loss on heating, max., per cent.....	...	...	54	54
	...	...	131	131
Flammability (burning rate), max.: Thickness, in.: Over 0.050, in. per min..... 0.031 to 0.050, sq. in. per sec.. 0.016 to 0.030, sq. in. per sec.. 0.011 to 0.015, sq. in. per sec.. 0.005 to 0.010, sq. in. per sec..	...	3.5 0.25 0.25 0.50 1.0	3.0 ...	2.5 ...
To be specified at a later date				

Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



(b) *Test Conditions*.—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) at  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the test methods or in these specifications.

(c) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designations: D 792).<sup>3</sup>

(d) *Tensile Strength and Elongation*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

(e) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Super-normal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

(f) *Haze Value*.—Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell (A.S.T.M. Designation: D 672).<sup>3</sup> Haze shall be measured on clear material only.

(g) *Water Absorption*.—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570).<sup>3</sup> using the 24-hr. immersion procedure. The test specimens shall be conditioned and reconditioned for 24 hr. at  $50 \pm 3$  C. ( $122 \pm 5$  F.).

(h) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648).<sup>3</sup> using a fiber stress of 264 psi.

(i) *Weight Loss on Heating*.—Test specimens 3 by 1 in. by the thickness of the sheet shall be conditioned for 48 hr. over anhydrous calcium chloride at  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall be weighed and then placed in a circulation air oven for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.). The specimens shall be

supported flatwise on a screen in the oven. Upon removal from the oven, the specimens shall be cooled in a desiccator over anhydrous calcium chloride to  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall then be weighed, and the percentage weight loss on heating calculated on the basis of the conditioned weight.

(j) *Flammability*.—Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568).<sup>3</sup> and the Standard Method of Test for Flammability of Plastics Over 0.050 in. in Thickness (A.S.T.M. Designation: D 635).<sup>3</sup>

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing the sheet which has been sampled according to Section 5. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the material may be rejected at the

option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### **Packing and Marking**

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure ac-

ceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name, stock number, and surface finish of the material, the size and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer and the number of the contract or order.

# Tentative Specifications for ETHYL CELLULOSE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 787 - 46 T

ISSUED, 1944; REVISED, JUNE 1946, SEPTEMBER 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover thermoplastic molding compounds consisting of ethyl cellulose and plasticizers, with or without the addition of dyes and pigments, and suitable for injection and extrusion molding. They do not include special materials compounded for special applications.

NOTE.—The properties included in these specifications are those required to identify the types and grades of molding compounds covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types and Grades

2. These specifications cover two types and seven grades of ethyl cellulose molding compounds, the grades being classified in accordance with their flow temperatures, as shown by Table I.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revisions accepted by the Society at annual meeting, June, 1946, and by the Administrative Committee on Standards, September 9, 1946.

## General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The size and form of granulations of the molding compound (Note), color,

TABLE I.—TYPES AND GRADES OF ETHYL CELLULOSE MOLDING COMPOUNDS.

Type	Grade	Flow Temperature	
		deg. Fahr.	deg. Cent.
I. General Purpose...	1.....	311 ± 9	155 ± 5
	2.....	293 ± 9	145 ± 5
	3.....	275 ± 9	135 ± 5
	4.....	257 ± 9	125 ± 5
II. Low-Temperature Resistant.....	5.....	311 ± 9	155 ± 5
	6.....	293 ± 9	145 ± 5
	7.....	275 ± 9	135 ± 5

and translucency or opacity shall be as specified by the purchaser in the contract or order.

NOTE.—Ethyl cellulose molding compounds are available in granular sizes of  $\frac{3}{16}$ ,  $\frac{1}{8}$ , and  $\frac{1}{4}$  in., and in other sizes and forms.

## Detail Requirements

4. Test specimens prepared under conditions specified by the manufacturer shall conform to the requirements as to

physical and electrical properties prescribed in Table II (Note).

NOTE.—Except for specially compounded material, ethyl cellulose molding materials are not designed for compression molding, and it is practically impossible to mold certain injection formulations by compression. The requirements in Table II are based upon extruded or injection molded test specimens.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Elec-

cellulose shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions*.—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the test method or in these specifications.

(c) *Flow Temperature*.—Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding

TABLE II.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

	Type I, General Purpose				Type II, Low-Temperature Resistant		
	Grade 1	Grade 2	Grade 3	Grade 4	Grade 5	Grade 6	Grade 7
Flow temperature {deg. Cent. $\pm 5$ .....	155	145	135	125	155	145	135
{deg. Fahr. $\pm 9$ .....	311	293	275	257	311	293	275
Tensile strength, min., psi. ....	5500	4000	3500	2500	4500	3000	2500
Specific gravity, 25/25 C. (77/77 F.), max. ....	1.14	1.15	1.15	1.16	1.14	1.12	1.09
Impact strength (Izod), min., {at 25 C. (77 F.) .....	2.5	3.5	3.5	3.0	5.0	5.0	5.0
{ft.-lb. per in. of notch {at -40 C. (-40 F.) .....	0.5	0.5	0.5	0.5	1.0	1.0	1.0
Heat distortion temperature at 264 {deg. Cent. ....	60	54	52	46	57	49	46
{psi. fiber stress, min. {deg. Fahr. ....	140	130	125	115	135	120	115
Water absorption (24-hr. immersion) {Weight gain plus soluble matter loss. ....	1.8	1.8	1.7	1.7	1.8	1.8	1.8
{Soluble matter loss .....	0.1	0.1	0.1	0.1	0.1	0.1	0.1
Weight loss on heating, max., per cent. ....	0.2	0.8	1.4	2.0	0.2	0.8	1.4

trical Insulators (A.S.T.M. Designation: D 392).<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens*.—For those tests where conditioning is required, molded test specimens of ethyl

Materials (A.S.T.M. Designation: D 569).<sup>3</sup> using the preferred method of conditioning.

(d) *Tensile Strength*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

(e) *Specific Gravity*.—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(f) *Impact Strength (Izod)*.—Method A of Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resist-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

ance of Plastics at Subnormal and Super-normal Temperatures (A.S.T.M. designation: D 758).<sup>3</sup>

(g) *Heat Distortion Temperature*. — Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using a fiber stress of 264 psi.

(h) *Water Absorption*. — Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570)<sup>3</sup> using the 24-hr. immersion procedure.

(i) *Weight Loss on Heating*. — Test specimens 2 in. in diameter by  $\frac{1}{8}$  in. in thickness shall be conditioned for 48 hr. over anhydrous calcium chloride at  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall be weighed and then placed in a circulation air oven for 72 hr. at  $82 \pm 1$  C. ( $180 \pm 2$  F.). The specimens shall be supported flatwise on a screen in the oven. Upon removal from the oven the specimens shall be cooled in a desiccator over anhydrous calcium chloride to  $25 \pm 1$  C. ( $77 \pm 2$  F.). The specimens shall then be weighed, and the percentage weight loss on heating calculated on the basis of the conditioned weight.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the

satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*. — The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation, at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*. — Shipping containers shall be marked with the name of the material, type, grade, and the quantity contained therein, as defined by the contract or order under which the shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Tentative Specifications for* METHACRYLATE MOLDING COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 788 - 44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover methacrylate molding compounds consisting of at least 51 per cent methyl methacrylate resin, and suitable for compression, injection, or extrusion molding.

NOTE.—The properties included in these specifications are those required to identify the types and grades of molding compounds covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

## Types

2. These specifications cover the following four types of methyl methacrylate molding compounds:

*Type I.*—A general purpose molding compound, for injection and extrusion molding, having a heat-distortion temperature between 57 and 71 C. (135 and 160 F.).

*Type II.*—A heat-resistant molding compound, for injection and extrusion molding, having a heat-distortion temperature between 71 and 88 C. (160 and 190 F.).

*Type III.*—A general purpose molding compound, for compression molding, having a heat-distortion temperature between 57 and 71 C. (135 and 160 F.).

*Type IV.*—A heat resistant molding compound, for compression molding, having a heat distortion temperature between 71 and 88 C. (160 and 190 F.).

## General Requirements

3. (a) The molding compounds shall be of uniform composition and so compounded as to conform to the requirements prescribed in these specifications.

(b) The size and form of granulations of the molding compound (Note), color, and translucency or opacity shall be as specified by the purchaser in the contract or order.

NOTE.—Types I and II clear, colored, and pigmented powders are usually supplied in granular sizes that will pass a No. 3 ( $\frac{1}{4}$ -in.) sieve.

Types III and IV clear powders are usually supplied in a pearl form having a particle size

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

distribution such that none will remain on a No. 10 (2000-micron) sieve and from 25 to 75 per cent will pass a No. 60 (250-micron) sieve.

Types III and IV colored and pigmented powders are usually supplied in granular sizes that will pass a No. 6 (3360-micron) sieve.

### Detail Requirements

4. Test specimens molded by compression or injection under conditions specified by the manufacturer (Note) shall conform to the requirements as to physical requirements prescribed in Table I.

and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—For those tests where conditioning is required, the molded test specimens of

TABLE I.—DETAIL REQUIREMENTS FOR MOLDED TEST SPECIMENS.

	Type I	Type II	Type III	Type IV	Remarks
Specific gravity, 25/25 C. (77/77 F.), max.....	1.2	1.2	1.2	1.2	Unpigmented and unfilled material
Index of refraction ( <i>n<sub>D</sub></i> ) {min. ....	1.48	1.48	1.48	1.48	Uncolored, unpigmented, and unfilled material
{max. ....	1.50	1.50	1.50	1.50	
Light transmission, min., per cent. ....	90	90	90	90	Uncolored, unpigmented, and unfilled material
Water absorption, 24-hr. immersion, max., per cent:					Unpigmented and unfilled material
Weight gain plus soluble matter loss .....	0.6	0.6	0.6	0.6	
Soluble matter loss .....	0.1	0.1	0.1	0.1	
Heat distortion temperature at {deg. Cent. 57		71	57	71	Unpigmented and unfilled material
264 psi. fiber stress, min. {deg. Fahr. 135		160	135	160	
Impact strength (Izod) {at 25 C. (77 F.) ....	0.2	0.2	0.1	0.1	Unpigmented and unfilled material
min., ft.-lb. per in. of notch {at -40 C. (-40 F.) ...	0.2	0.2	0.1	0.1	
{deg. Cent. 155		175	155	175	
Flow temperature, max. {deg. Fahr. ....	311	347	311	347	

NOTE.—All test specimens shall be molded to conform with the applicable procedures described in methods of the American Society for Testing Materials.

### Sampling

5. The molding compound shall be sampled in accordance with the sampling procedure described in the Standard Method of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation D 392).<sup>3</sup> Method B of Standard D 392 shall be used in determining particle size distribution. A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment

methacrylate shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity unless otherwise specified in the test methods or in these specifications.

(c) *Specific Gravity.*—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(d) *Index of Refraction.*—Standard Methods of Test for Index of Refraction

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



of Transparent Organic Plastics (A.S.T.M. Designation: D 542).<sup>3</sup>

(e) *Light Transmission*.—Tentative Method of Test for Luminous Reflectance and Transmission Characteristics and Color of Plastic Materials (A.S.T.M. Designation: D 791).<sup>3</sup>

(f) *Water Absorption*. — Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion procedure.

(g) *Heat Distortion Temperature*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using a fiber stress of 264 psi.

(h) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

(i) *Flow Temperature*. — Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (A.S.T.M. Designation: D 569),<sup>3</sup> using the alternative method of conditioning.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these specifica-

tions. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

# *Tentative Specifications for*

## NYLON INJECTION MOLDING COMPOUND<sup>1</sup>



A.S.T.M. Designation: D 789 - 44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover a molding compound consisting of nylon with or without the addition of dyes, pigments, plasticizers, modifiers, and lubricants, and suitable for injection molding.

NOTE.—The properties included in these specifications are those required to identify the type of molding powder covered. There may be other requirements necessary to identify particular characteristics. These will be added to these specifications as their inclusion becomes desirable and the necessary test data and methods become available.

### Types and Grades

2. These specifications cover one type and one grade of nylon molding powder intended specifically for injection molding.

### General Requirements

3. (a) The molding powder shall be of uniform composition and so prepared as to conform to the requirements prescribed in these specifications.

(b) The size and form of granulations of the molding powder (Note) shall be as specified by the purchaser in the contract or order.

NOTE.—Nylon molding powders are available in particle sizes of  $\frac{1}{8}$  by  $\frac{1}{4}$  by  $\frac{1}{2}$  in.; in granulations of which 98 per cent will pass a  $\frac{1}{8}$  in. sieve and be retained on a  $\frac{1}{2}$  in. sieve; and in fine particles less than  $\frac{1}{16}$  in. in largest dimension.

(c) The molding powder, when tested within three weeks of receipt by the purchaser, shall contain not more than 0.3 per cent of moisture (Note).

NOTE.—It is essential that the moisture content of the powder at the time of molding be less than 0.3 per cent, since otherwise the molded articles will be inferior in appearance and in physical properties, even though satisfactory for some purposes. Drying under atmospheric pressure will not yield a powder of optimum properties, even though molded articles of satisfactory appearance may be made from powder dried at a temperature of 100 C. for not longer than 3 hr. A higher temperature or a longer exposure causes deterioration.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

### Detail Requirements

4. Test specimens molded by injection, under conditions specified by the manufacturer, shall conform to the following requirements as to physical and electrical properties:

Specific gravity, 25/25 C. (77/77 F.) (unpigmented), max.....	1.15
Heat distortion temperature, min.:	
at 66 psi. fiber stress.....	193 C. 380 F.
at 264 psi. fiber stress.....	74 C. 165 F.
Deformation under load of 4000 psi. at 50 C. (122 F.), max., per cent....	5
Impact strength (Izod), min., ft.-lb.	
(at -40 C. (-40 F.)	0.45
per in. of notch at 25 C. (77 F.)....	0.6
(at 60 C. (140 F.)....	0.9
Water absorption (24-hr. immersion), max., per cent.....	1.5
Tensile strength, (at -40 C. (-40 F.)	14 000
min., psi. .... (at 25 C. (77 F.)....	9 000
(at 60 C. (140 F.)....	8 000
Mandrel-bend number, min., per cent. { regular granulation.....	90
{ 1/16-in. granulation.....	85
Dielectric strength (short-time test), min., v. per mil.....	350

### Sampling

5. The molding powder shall be sampled in accordance with the sampling procedure described in the Standard Methods of Testing Molding Powders Used in Manufacturing Molded Electrical Insulators (A.S.T.M. Designation: D 392).<sup>3</sup> A batch of molding compound shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

#### (a) Conditioning Test Specimens.—

For those tests where conditioning is required, the molded test specimens of nylon shall be conditioned in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—Tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity, unless otherwise specified in the test methods or in these specifications.

(c) *Specific Gravity.*—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(d) *Heat Distortion Temperature.*—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648),<sup>3</sup> using fiber stresses of 66 and 264 psi. as specified.

(e) *Deformation Under Load.*—Tentative Methods of Test for Deformation of Plastics Under Load (A.S.T.M. Designation: D 621),<sup>3</sup> using Method A, 1000-lb. load.

(f) *Impact Strength (Izod).*—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256)<sup>3</sup> and the Tentative Method of Test for Impact Resistance of Plastics at Subnormal and Supernormal Temperatures (A.S.T.M. Designation: D 758).<sup>3</sup>

(g) *Water Absorption.*—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion period.

(h) *Tensile Strength.*—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638)<sup>3</sup> and the Tentative Methods of Test

<sup>3</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

for Tensile and Compressive Properties of Plastics at Subnormal and Super-normal Temperatures (A.S.T.M. Designation: D 759).<sup>3</sup>

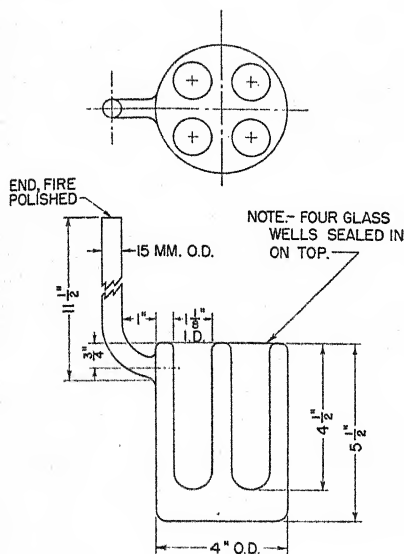


FIG. 1.—Diphenylene Oxide Bath (Pyrex).

(i) *Moisture Content*.—The moisture content of nylon molding powder shall be determined as follows:

(1) *APPARATUS*.—Test tubes, 6 by 1 in.;<sup>4</sup> lime-glass test tubes, 5 by 0.625 in.; No. 11 cork stoppers, No. 5 neoprene stoppers, each carrying a 2.5-in. glass tube with 4-mm. bore; desiccator charged with anhydrous phosphorus pentoxide; Hyvac pump or equivalent; mercury type manometer; diphenylene oxide bath (see Fig. 1); and diphenylene oxide in a melting range of 81 to 84 C (178 to 183 F.).(Note).

NOTE.—When the diphenylene oxide is boiling, the level should be approximately 0.25 in. below the bottom of the wells of the bath. Its boiling point should be 180 C. (356 F.).

(2) *PROCEDURE*.—Place a 5 by 0.625-in. lime-glass test tube inside a 6 by 1-in. tube and dry for 5 to 10 min. in

one of the wells of the diphenylene oxide bath which shall be refluxing half way up the air condenser. Cool in the desiccator and weigh to the nearest 1 mg. Fill the small test tube with molding powder to about 2 in. from the top, replace in the larger tube and reweigh. Attach the larger test tube to the vacuum with the neoprene stopper, and evacuate in the cold for 1 to 2 min. Reduce the pressure to between 1 to 5 mm. before replacing the tube in one of the wells of the bath. After heating for 20 min., remove the tube from the bath and allow to cool for 7 min. under vacuum. Carefully release the vacuum so as not to break the manometer. Remove the stopper and cork the larger tube to prevent glass, shattered as the nylon cools, from flying out of the tube. Allow the tube to cool in air and while it is still warm place it in the desiccator. When finally cooled, remove the cork and carefully brush any adhering glass into the tube. Weigh the tube and contents to the nearest 1 mg.

(3) *CALCULATION*.—Calculate the moisture content as follows:

Moisture, per cent =

$$\frac{\text{Loss in wt.}}{\text{Original wt. of sample}} \times 100$$

(j) *Dielectric Strength*.—Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149),<sup>3</sup> using the short-time test in oil. The test specimen shall be 6 by 6 by  $\frac{1}{8}$  in. and shall be conditioned for 48 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity.

(k) *Mandrel Bend*.—The mandrel-bend test to measure the ability of standard  $\frac{1}{8}$ -in. impact specimens to sustain bending around a  $\frac{1}{4}$ -in. mandrel shall be made as follows:

<sup>4</sup> Pyrex glass tubes are recommended.

(1) APPARATUS.—A hinged mandrel-bend tester as illustrated in Fig. 2.

(2) PROCEDURE.—Condition 25 test specimens 5 by 0.5 by 0.125 in. in the dry condition by sealing them for 24 hr. prior to test in an atmosphere of 0 to 5 per cent relative humidity immediately on removal from the injection machine. A rubber-gasketed glass jar may conveniently be used as the receptacle for the specimens. Slide each specimen through the slot in the tester and bend it double upon itself around the  $\frac{1}{8}$ -in. mandrel. Make the test at

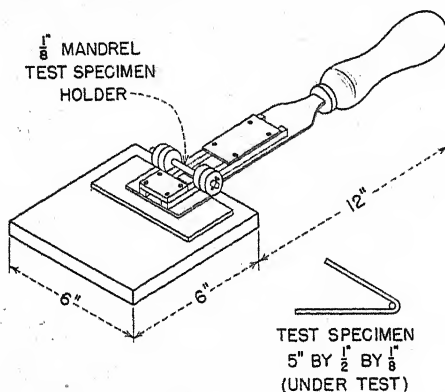


FIG. 2.—Mandrel-Bend Tester.

four places on each specimen, thus providing a total of 100 bends from the 25 specimens.

(3) CALCULATION.<sup>5</sup>—Calculate the mandrel-bend number as follows:

$$\text{Mandrel-bend number} = 100 - \text{number of breaks}$$

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed

in Section 6 shall be used to establish conformity of a material to these specifications. It is recommended that routine inspection be limited to those tests required to identify the material to the satisfaction of the purchaser. The purchaser shall state in the contract or order the tests which the manufacturer will be required to make on each shipment for identification of the material.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the batch of molding compound may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers of a type which will keep the moisture constant at a point less than 0.3 per cent. All containers shall be so constructed as to ensure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, and quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer and the number of the contract or order.

<sup>5</sup> The formula for calculating mandrel-bend number was editorially corrected in October, 1946.

# Tentative Specifications for

## CAST ALLYL PLASTIC SHEETS, RODS, TUBES, AND SHAPES<sup>1</sup>



A.S.T.M. Designation: D 819 - 45 T

ISSUED, 1945.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover allyl plastic sheets, rods, tubes, and other shapes produced by casting or machining cast blanks. These specifications do not apply to heat-formed parts and shapes or sections that are made by assembling two or more pieces.

NOTE.—The properties included in these specifications are those required to identify the types and grades of materials covered. There may be other requirements necessary to identify particular characteristics. These will be added to the specifications as their inclusion becomes generally desirable and the necessary test data and methods become available.

### Types and Grades

2. These specifications cover one type of cast allyl plastics: namely, a general purpose material.

### General Requirements

3. The size, shape, dimensional tolerances, surface finish, and color shall be as specified by the purchaser in the contract or order (Note).

NOTE.—Sheet material is available in the following standard sizes:

Width, in.....	45
Length, in.....	57
Nominal thickness, in.....	$\frac{1}{16}$ , $\frac{3}{32}$ , $\frac{1}{8}$ , $\frac{3}{16}$ , $\frac{1}{4}$ , $\frac{5}{16}$ , $\frac{3}{8}$ , $\frac{1}{2}$

Sheet material is subject to permissible variations of plus or minus  $\frac{1}{16}$  in. in width and length, and to the following permissible variations in thickness from the specified dimensions:

Nominal Thickness, in.	Permissible Variations, <sup>a</sup> in.	
	Under	Over
$\frac{1}{16}$ .....	0.012	0.020
$\frac{3}{32}$ .....	0.011	0.018
$\frac{1}{8}$ .....	0.015	0.023
$\frac{3}{16}$ .....	0.022	0.022
$\frac{1}{4}$ .....	0.030	0.038
$\frac{5}{16}$ .....	0.034	0.046
$\frac{3}{8}$ .....	0.040	0.045
$\frac{1}{2}$ .....	0.055	0.060

<sup>a</sup> These permissible variations apply at any point on the surface of the sheet.

Rods and tubes are subject to the following permissible variations from the specified dimensions: Plus or minus  $\frac{1}{16}$  in. in length, plus or minus 10 per cent in outside diameter, and plus or minus 5 per cent in inside diameter.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Accepted by Committee E - 10 on Standards, May 10, 1945.

### Detail Requirements

4. The material shall conform to the requirements prescribed in Table I.

### Sampling

5. A sample shall be selected from sheet in thickness sufficient to determine conformance of the material to these specifications. In the case of materials that are normally processed only into special sizes and shapes from which the required test specimens cannot be cut, the test specimens shall be prepared specially from the material under conditions simulating those of the manufacture of the special size or shape.

TABLE I.—DETAIL REQUIREMENTS FOR CAST ALLYL RESIN PLASTICS.

Index of refraction, $n_D$ .....	$\left\{ \begin{array}{l} \text{min.} \\ \text{max.} \end{array} \right.$	$\left\{ \begin{array}{l} 1.49 \\ 1.51 \end{array} \right.$
Specific gravity 25/4 C. (77/39 F.).....	$\left\{ \begin{array}{l} \text{min.} \\ \text{max.} \end{array} \right.$	$\left\{ \begin{array}{l} 1.30 \\ 1.32 \end{array} \right.$
Light transmission, min., per cent.....		91
Haze, max., per cent.....		3.0
Displacement factor (optical), max.....		25 <sup>a</sup>
Water absorption:		
Gain in weight, max., per cent.....		0.4
Soluble matter loss, max., per cent....		0.1
Acetone absorption:		
Gain in weight, max., per cent.....		0.5
Heat distortion deflection at 130 C (266 F.), max., mils.....		80
Heat distortion temperature at 264 psi. fiber stress.....	$\left\{ \begin{array}{l} \text{min.} \\ \text{max.} \end{array} \right.$	$\left\{ \begin{array}{l} 60 \\ 80 \end{array} \right.$
Mar resistance, min., per cent (1200 g.)..		90
Tensile strength, min., psi.....		5000
Impact strength (izod), min., ft.-lb. per in. of notch.....		0.3

<sup>a</sup> A displacement factor of 25 is equivalent to 7 min. of arc or  $\frac{1}{8}$  in. image displacement at 25 ft.

### Methods of Testing

6. The properties enumerated in these specifications shall be determined in accordance with the following methods of the American Society for Testing Materials:

(a) *Conditioning Test Specimens.*—For those tests where conditioning is required, the test specimens of allyl plastics shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions.*—The tests shall be conducted in the standard laboratory atmosphere of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity; unless otherwise specified in the testing methods or in these specifications.

(c) *Index of Refraction.*—Standard Methods of Test for Index of Refraction of Transparent Organic Plastics (A.S.T.M. Designation: D 542),<sup>3</sup> using one of the exposed surfaces of the test specimen that has been given any necessary polish without gross removal of material.

(d) *Specific Gravity.*—Method A of the Tentative Methods of Test for Specific Gravity of Plastics (A.S.T.M. Designation: D 792).<sup>3</sup>

(e) *Light Transmission.*—Tentative Method of Test for Luminous Reflectance and Transmission Characteristics and Color of Plastic Materials (A.S.T.M. Designation: D 791).<sup>3</sup>

(f) *Haze.*—Tentative Method of Test for Haze of Transparent Plastics by Photoelectric Cell (A.S.T.M. Designation: D 672),<sup>3</sup> except that the test specimens shall have a thickness not over  $\frac{1}{2}$  in.

(g) *Displacement Factor.*—Standard Method of Test for Surface Irregularities of Flat Transparent Plastic Sheet (A.S.T.M. Designation: D 637),<sup>3</sup> using test specimens consisting of flat sheets 18 by 18 in.

(h) *Water Absorption.*—Standard Method of Test for Water Absorption of Plastics (A.S.T.M. Designation: D 570),<sup>3</sup> using the 24-hr. immersion procedure and test specimens  $\frac{1}{8}$  in. in thickness. The test specimens shall be conditioned and reconditioned for 24 hr. at  $50 \pm 3$  C. ( $122 \pm 5$  F.).

(i) *Acetone Absorption.*—Standard Method of Test for Resistance of Plastics to Chemical Reagents (A.S.T.M. Designation: D 543).<sup>3</sup> Only the acetone reagent shall be used and only the weight change of the sample shall be determined.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



(j) *Heat Distortion*.—Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648).<sup>3</sup> The test specimens shall be taken from material as received. Any square, rectangular, or cylindrical test specimen may be used but the test load used shall be calculated on the basis of a center-loaded beam in which the outer fiber stress produced is 264 psi. Pieces of thin sheets  $\frac{1}{2}$  in. in width may be stacked and bound together to form a test specimen approximately  $\frac{1}{2}$  by  $\frac{1}{2}$  in. If such a specimen is used, it shall be loaded parallel to the width of the assembled pieces. The test shall be continued to 130 C. (266 F.) and the amount of distortion between 25 and 130 C. (77 and 266 F.) shall be recorded.

(k) *Mar Resistance*.—Standard Method of Test for Mar Resistance of Plastics (A.S.T.M. Designation: D 673).<sup>3</sup> The test shall be made with 1200 g. of emery.

(l) *Tensile Strength*.—Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup> The test specimens shall be taken from material as received and determinations made on at least five specimens and the results averaged.

(m) *Impact Strength (Izod)*.—Method A of the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup> The test specimen shall be taken from material as received.

#### Number of Tests

7. One set of test specimens as prescribed in the methods of testing (Section 6) shall be considered sufficient for testing each batch. The average result for the specimens tested shall conform to the requirements prescribed in these specifications. All of the tests listed in Section 6 shall be used to establish conformity of a material to these speci-

cations. It is recommended that routine inspection be limited to the following:

#### *For Sheets:*

Thickness and Dimensions,  
Appearance, and  
Displacement Factor.

#### *For Rods and Tubes:*

All dimensions, and  
Appearance.

#### Retest and Rejection

8. If the results of any test do not conform to the requirements prescribed in these specifications, at the option of the manufacturer that test shall be repeated on two additional sets of specimens from the same batch or shipment, each of which shall conform to the requirements specified. If either of these two additional sets of specimens fails, the material may be rejected at the option of the purchaser. Notice of failure of material based on tests made in accordance with these specifications shall be reported to the manufacturer within three weeks from the receipt of the material by the purchaser. Any material which it is claimed has failed to conform to the requirements of these specifications shall be retained in the original packages until proper disposition has been agreed upon by the manufacturer and the purchaser.

#### Packing and Marking

9. (a) *Packing*.—The material shall be packed in standard commercial containers, so constructed as to insure acceptance by common or other carriers for safe transportation at the lowest rate to the point of delivery, unless otherwise specified in the contract or order.

(b) *Marking*.—Shipping containers shall be marked with the name of the material, type, grade, form, and the quantity contained therein, as defined by the contract or order under which shipment is made, the name of the manufacturer, and the number of the contract or order.

*Tentative Specifications for*  
**MOLDS FOR TEST SPECIMENS OF MOLDING MATERIALS  
USED FOR ELECTRICAL INSULATION<sup>1</sup>**



**A.S.T.M. Designation: D 647 - 45 T**

**ISSUED, 1941; REVISED, 1942, 1945.<sup>2</sup>**

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Scope**

1. These specifications cover the design of standard molds to be used to mold test specimens from molding materials used for electrical insulation. The press and molding cycle used in molding test specimens should be agreed upon by the purchaser and the seller of the material.

**NOTE.**—The design of any molds not yet covered by these specifications should be agreed upon by the seller of the material and the purchaser, in order to obtain concordant results.

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<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Latest revision accepted by the Administrative Committee on Standards, December 8, 1945.

**Molds for  $\frac{1}{2}$  by  $\frac{1}{2}$  by 5 in. Specimen**

2. Molds for the  $\frac{1}{2}$  by  $\frac{1}{2}$  by 5 in. or the  $\frac{1}{2}$  by  $\frac{1}{2}$  by 2 $\frac{1}{2}$  in. test specimens may be the single bar, single cavity positive mold type and shall conform to the design and dimensions shown in Fig. 1.<sup>3</sup>

**Molds for Disk Test Specimen**

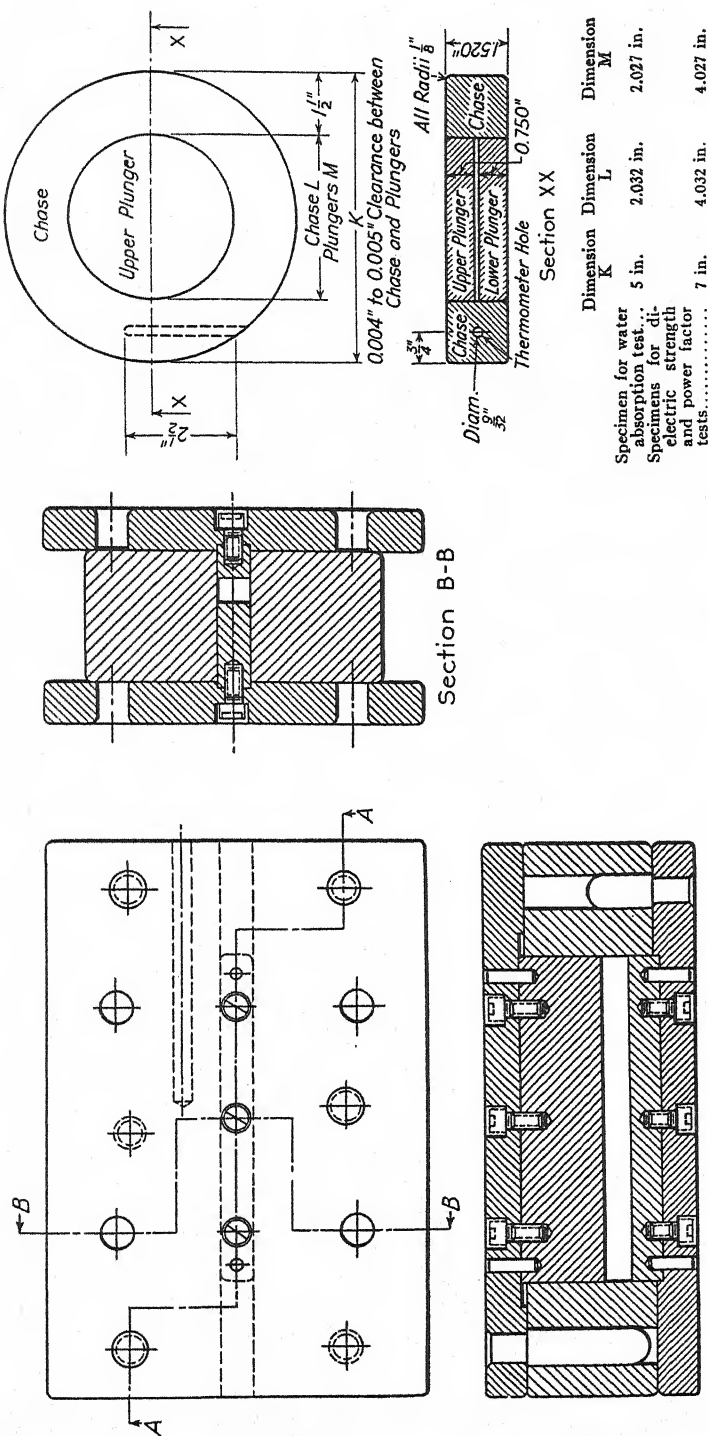
3. Molds for the disk test specimens 2 in. or 4 in. in diameter shall be of the design and dimensions shown in Fig. 2.

**Molds for Tension Test Specimen**

4. Molds for the tension test specimen shall be of the design and dimensions shown in Fig. 3.<sup>3</sup>

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<sup>3</sup> Blueprints of detailed drawings for the construction of the molds shown in Figs. 1 and 3 are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.



Section A-A

FIG. 1.—Single Bar, Single Cavity Positive Mold for  $\frac{1}{2}$  by  $\frac{1}{2}$  by  $\frac{1}{2}$  in. Test Specimen.<sup>3</sup>

FIG. 2.—Mold for Disk Test Specimens.

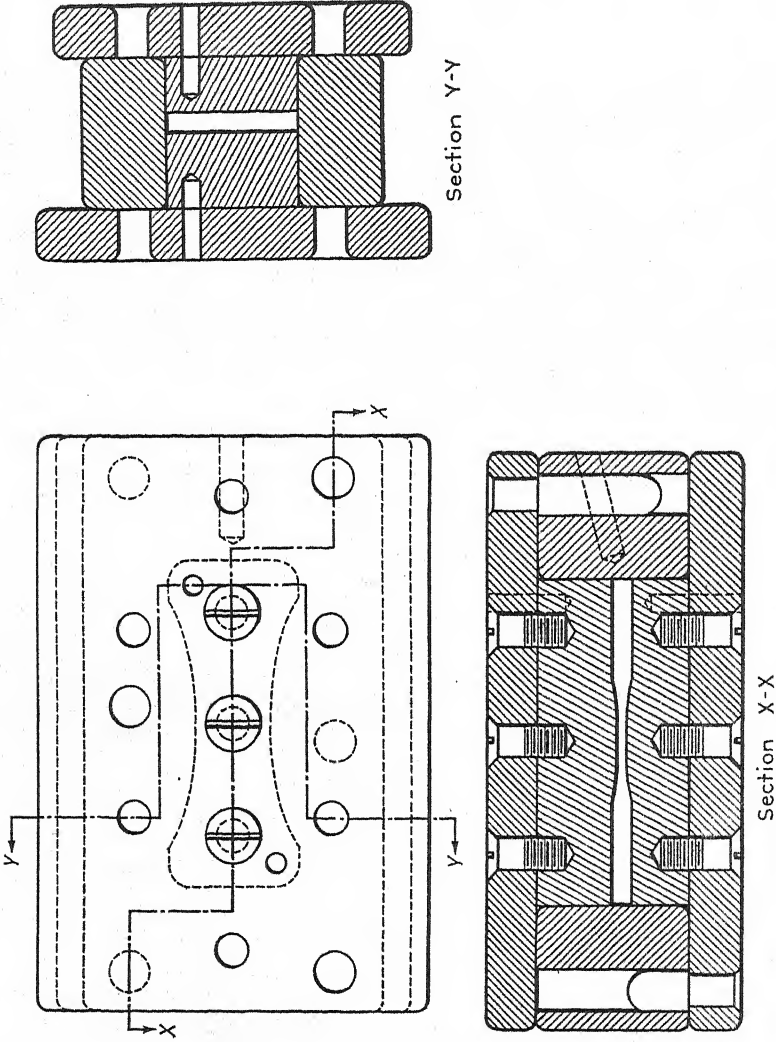


FIG. 3.—Mold for Tension Test Specimen.<sup>a</sup>

# *Tentative Method of Test for* ACETYL AND BUTYRYL CONTENT OF CELLULOSE ACETATE BUTYRATE<sup>1</sup>



A.S.T.M. Designation: D 817 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. This method covers the procedure for determining by chemical analysis the acetyl and butyryl content of cellulose acetate butyrate.

## Outline of Method

2. The total acyl content of the ester is determined, a mixture of the combined acids is isolated, and the molar ratio of the acids in the mixture is determined on the basis of the distribution ratios of the acids between water and *n*-butyl acetate. The distribution ratios are determined for the individual acids, using samples of known high purity, and from these values and the distribution ratios of the mixed acids the molar ratios of the acids are calculated. From the total acyl content and the molar ratios of the acids the percentages by weight of acetyl and butyryl are calculated.

<sup>1</sup>Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on plastics.

This method of analysis is covered by U. S. Patent No. 2,069,892 owned by Eastman Kodak Company. However, in order to promote the better understanding of cellulose esters containing a plurality of fatty acid groups of 2 to 4 carbon atoms, such as cellulose acetate propionate and cellulose acetate butyrate, Eastman Kodak Company grants a royalty-free, non-exclusive license to use the method of analysis defined by its U. S. Patent No. 2,069,892 for the full term of that patent, provided that such license shall not extend to the analysis of esters which, or the process or apparatus for making which, are in violation of any other patent owned or controlled by it.

<sup>2</sup>Accepted by Committee E-10 on Standards, December 11, 1944.

## Apparatus

3. *Vacuum Distillation Apparatus.*—The vacuum distillation apparatus shown in Fig. 1 will be required. The 500-ml. round-bottom flask *A* shall be fitted with a stopper carrying a very small capillary inlet tube *B* and a Kjeldahl distilling head *C*. The Kjeldahl distilling head shall be connected to a vertical condenser *D* having an outlet tube long enough to reach within 3 in. of the bottom of the 500-ml. distilling flask *E* used as a receiver. The Kjeldahl distilling head shall be equipped with a funnel *F* for adding extra water during the distillation. A water bath *G* for heating the sample and a cooling bath *H* for cooling the receiver shall be provided.

## Reagents

4. (a) *Ethyl Alcohol* (75 per cent).
- (b) *Sodium Hydroxide Solution* (approximately 1 N).
- (c) *Standard Sodium Hydroxide Solution* (0.5 N).
- (d) *Standard Sodium Hydroxide Solution* (0.1 N).
- (e) *Standard Hydrochloric Acid* (0.5 N).
- (f) *Acetone.*—Acetone neutral with respect to pH.

(g) *Phosphoric Acid (1 M).*—Dilute 68 ml. of  $H_3PO_4$  (85 per cent) to 1 liter with distilled water.

(h) *n-Butyl Acetate.*—Prepare *n*-butyl acetate, for use as an extraction solvent,

nearly free of acid and alcohol as possible), add about 0.5 lb. of anhydrous  $Na_2CO_3$  and about 0.5 lb. of anhydrous  $Na_2SO_4$ . This is preferably done in a 5-gal. bottle. Agitate the solution thoroughly, and allow to settle for 24 hr. or longer. For use, siphon off the clear liquid and filter. It is best to draw off only what will be used in a day or two and leave the remainder in the stock bottle over the dehydrating salt.

Check for acidity by shaking 60 ml. of the *n*-butyl acetate with 30 ml. of distilled water in a 125-ml. separatory funnel for about 1 min. Allow to settle, draw off the water layer, and titrate with 0.1 *N* NaOH, using phenolphthalein as the indicator. If this requires more than 0.02 ml. of 0.1 *N* NaOH, the solution should be reagentized and checked again.

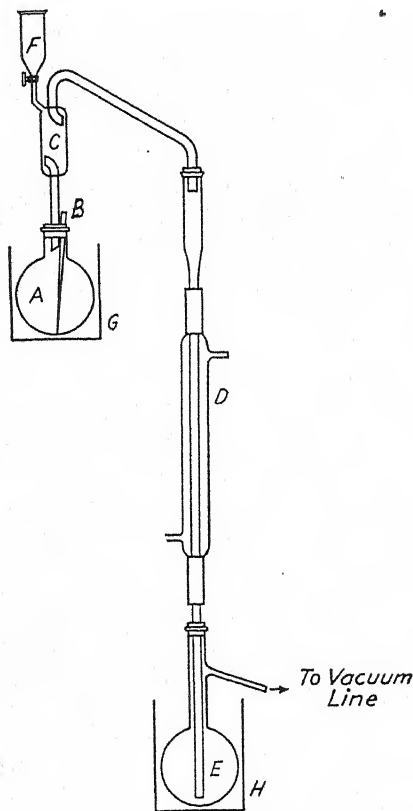
(i) *Acetic and Butyric Acids.*—Acetic and butyric acids of tested purity.

### Determination of Total Acyl Content<sup>3</sup>

5. (a) If the sample is not in a soft fluffy form, grind or powder it.<sup>4</sup> Dry the sample to constant weight (about 2 hr.) at 100 to 105 C. and cool in a desiccator.

(b) Place duplicate 1-g. portions of the sample, *accurately* weighed on an analytical balance, in Erlenmeyer flasks, add 40 ml. of ethyl alcohol (75 per cent) to each flask, and stopper the flasks. Heat for 30 min. at about 55 C. in a water bath or an oven to swell the fibers. Add 40 ml. of 0.5 *N* NaOH, accurately measured from a burette, to each flask, stopper the flasks loosely, and heat them at about 55 C. for 15 min. Tightly stopper the flasks and allow them to stand for two days at room temperature.

(c) Back-titrate the excess NaOH with standard 0.5 *N* HCl, using phenolphthalein as the indicator. After reaching a temporary end point, allow the flask to stand at least 30 min. so the NaOH can diffuse from the fibers. Add 0.5 *N* HCl at intervals until all the free NaOH has been neutralized (Note). Great care must be taken at this point, or poor check values will be obtained.



A—Flask containing sample (500-ml., round-bottom).  
B—Capillary inlet tube.  
C—Kjeldahl distilling head.  
D—Condenser.  
E—Receiver (500-ml. distilling flask).  
F—Funnel for adding water.  
G—Water bath for heating sample.  
H—Cooling bath for receiver.

FIG. 1.—Vacuum Distillation Apparatus for Mixed-Ester Analysis.

free of acidity and water and containing little, if any, butyl alcohol.

NOTE.—A convenient method of preparing butyl acetate for use is as follows:

To 5 gal. of *n*-butyl acetate (90 to 100 per cent as received from the producers, and as

<sup>3</sup>All glassware should be chemically resistant.

<sup>4</sup>A Wiley mill is suitable for this purpose

NOTE.—If unsatisfactory results are obtained and there is evidence that the alkali is not reacting properly due to the nature of the precipitate, the material may be dissolved in a solvent such as acetone and reprecipitated into a fluffy form, or the alternative procedure for determination of total acyl content described in Section 6 may be used.

(d) Calculate the total acyl content as percentage apparent acetyl as follows:

Apparent acetyl, per cent =

$$\frac{VN - V_1N_1}{W} \times 4.302$$

where:

$V$  = milliliters of NaOH solution required for titration of the sample,

$N$  = normality of the NaOH solution,

$V_1$  = milliliters of HCl required for titration of the sample,

$N_1$  = normality of the HCl, and

$W$  = grams of sample used.

(e) If duplicate determinations differ by more than 0.5 per cent apparent acetyl, the analysis shall be repeated.

#### Alternative Determination of Total Acyl Content<sup>3</sup>

6. (a) Weigh approximately 1.5 g. of the sample into a glass weighing bottle. Dry the sample to constant weight (about 2 hr.) at 100 to 105 C. Cool in a desiccator and weigh *accurately*. Transfer to a 300-ml. Erlenmeyer flask, and weigh the empty bottle to obtain the weight of sample taken by difference.

(b) Add 5 ml. of distilled water and allow the sample to become thoroughly wet. Add 50 ml. of neutral acetone to the flask and close with a rubber stopper. Do not allow the solution to come in contact with the stopper. Place the flask in a shaking machine as soon as the acetone has been added, so that the sample will dissolve readily without balling up.

(c) When solution is complete, remove the stopper carefully and add slowly 50 ml. of 0.5  $N$  NaOH (use temperature correction on the burette), while swirling the flask. The cellulose acetate butyrate should precipitate in very fine particles, free of lumps. Place the flask in the shaker for 1 hr. at 20 C. At the end of this time remove the flask from the shaker, loosen the stopper, and allow it to drain into the flask for 2 or 3 min. Add phenolphthalein indicator and titrate with 0.5  $N$  HCl (use temperature correction on the burette).

(d) When the titration nears the end point, as evidenced by fading of the pink color from the solution, add approximately 1 ml. more of the 0.5  $N$  HCl to go past the end point. Shake vigorously for 2 min. Back-titrate with 0.5  $N$  NaOH to a *faint pink* end point, adding 1 drop of 0.5  $N$  HCl, which should fade out the slight pink. Add the amount of NaOH used in this titration to the original amount of NaOH used in starting the test (Paragraph (c)).

(e) Calculate the total acetyl content as percentage apparent acetyl in accordance with Section 5 (d). If duplicate determinations differ by more than 0.5 per cent apparent acetyl, the analysis shall be repeated.

#### Isolation of the Mixed Acids

7. (a) Heat duplicate 3-g. portions of the sample, not especially dried nor accurately weighed, with 60 ml. of approximately 1  $N$  NaOH in 500-ml., round-bottom, chemically resistant glass flasks in a water bath at 40 C. for 48 to 72 hr. At the end of this time add 60 ml. of 1  $M$   $H_3PO_4$  solution to each flask (enough to form monosodium phosphate, which liberates the organic acids from their sodium salts).

(b) Assemble the vacuum distillation apparatus as illustrated in Fig. 1. Heat



the 500-ml. round-bottom flask containing the sample in a water bath, and vacuum distill the acid solutions to dryness, allowing a small stream of air bubbles to enter to avoid bumping. Keep the receiver cooled to 0 C. Add 25 ml. of distilled water to the residue in each flask and again distill to dryness. Repeat the distillation to dryness with a second 25-ml. portion of distilled water.

NOTE.—In this operation it is not necessary to work with quantitative accuracy at all stages, but it is necessary to obtain water solutions of the acids in the same ratios as they occur in the esters. The volume of the distillate and rinsings is usually 200 to 250 ml., which in the majority of cases automatically adjusts the acidity of the distillate to from 0.06 to 0.12 *N*, the range desired for subsequent extractions.

#### Determination of the Molar Ratios of the Acids

8. (a) Titrate a 25-ml. portion of the distillate (Section 7 (b)) with 0.1 *N* NaOH, using phenolphthalein as the indicator. Designate the volume of NaOH required as *M*. Shake 30 ml. of the distillate in a small separatory funnel with 15 ml. of *n*-butyl acetate. Measure these volumes accurately using pipettes and burettes. Shake the mixture thoroughly for 1 min., allow the layers to separate for 2 min., and draw off the aqueous (lower) layer. Pipette out 25 ml. of the solution and titrate against 0.1 *N* NaOH (Note 1). Designate the volume of NaOH required as *M*<sub>1</sub>. Calculate *K*, the percentage distribution ratio of the acids in the distillate, as follows:

$$K = \frac{M_1}{M} \times 100$$

NOTE 1.—It should be kept in mind that all these determinations are ratios and not quantitative; however, accuracy of duplication is very important. All measurements must be made as exactly as those made by standardizations of the solutions and equipment.

(b) In the same manner determine the distribution ratios for acetic and butyric acids. Dilute a sample of each acid of tested purity with distilled water to give an approximately 0.1 *N* solution. Titrate 25-ml. portions and extract 30-ml. portions, following exactly the same procedure as used for the mixtures (Paragraph (a)). Calculate the distribution ratios for the pure acids, as decimal fractions, as follows (Note 2):

$$k = \frac{M_1}{M}$$

where:

*k*<sub>a</sub> = distribution ratio for acetic acid under the conditions described, and

*k*<sub>b</sub> = distribution ratio for butyric acid under the conditions described.

NOTE 2.—The constants must be checked occasionally and must be determined by each operator for each supply of butyl acetate. Blanks should be run on the butyl acetate, since it may develop acidity on standing, particularly if it contains a little water. All measurements should be made with good pipettes or burettes, and extreme care and cleanliness observed during the whole operation. The accuracy of the procedure can be checked by testing an acid mixture of known composition.

(c) *Calculation of Molar Ratios of the Acids.*—Calculate the molar ratios of butyric and acetic acids in the mixed acids as follows (Note 3):

$$B = \frac{K - 100k_a}{k_b - k_a}$$

$$A = 100 - B$$

where:

*B* = mole percentage of butyric acid,

*K* = percentage distribution ratio of the acids in the distillate (Paragraph (a)),

*k*<sub>a</sub> = distribution ratio of acetic acid (Paragraph (b)),

*k*<sub>b</sub> = distribution ratio of butyric acid (Paragraph (b)), and

*A* = mole percentage of acetic acid.

NOTE 3.—In order to evaluate two unknowns, two simultaneous algebraic equations involving the two unknown quantities are necessary. In the case of a binary acid mixture, the sum of the mole percentages of the acids present represents the total acidity, or 100 per cent. If  $A$  and  $B$  represent the mole percentages of acetic and butyric acids respectively:

$$\begin{aligned} A + B &= 100 \\ Ak_a + Bk_b &= K \end{aligned}$$

The distribution ratios  $k_a$  and  $k_b$  are known and refer to the pure individual acids, whereas the distribution ratio  $K$  refers to the binary mixture. By solving these equations for  $B$ , the formulas given in Paragraph (c) may be derived.

### Calculation of Acetyl and Butyryl Content

9. Calculate the percentages by weight of acetyl and butyryl as follows:

$$\text{Acetyl, per cent} = AC$$

$$\text{Butyryl, per cent} = BC \times \frac{71}{43}$$

where:

$A$  = mole percentage of acetic acid (Section 8),

$B$  = mole percentage of butyric acid (Section 8), and

$C$  = percentage by weight of apparent acetyl (Section 5 or Section 6).

# *Tentative Method of Test for*

## AMMONIA IN PHENOL-FORMALDEHYDE MOLDED MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 834 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers the determination of ammonia in phenol-formaldehyde molded parts and molding powders.

### Apparatus

2. (a) *Nessler Tubes*.—Nessler tubes of 50-ml. capacity (either tall- or short-form).

(b) *Nesslerimeter*.

(c) *Balance*.—A balance sensitive to at least 0.01 g.

(d) *Pipettes*.—Pipettes of suitable capacities, including 100, 10, and 2 ml.

(e) *Flask*.—A 250-ml. glass-stoppered flask.

(f) *Distillation Flask*.—A 100-ml. Claissen-type distillation flask.

(g) *Condenser*.

(h) *Crucible*.—A sintered-glass crucible of medium porosity (conveniently of 30-ml. capacity), or equivalent.

(i) *Bunsen Burner*.

(j) *Porous Plate*.—Chips of porous plate.

### Reagents

3. (a) *Ammonia-Free Distilled Water*.—Boil distilled water in a glass vessel for 2 min., and cool.

(b) *Potassium Permanganate, c.p.*—Either solid  $\text{KMnO}_4$  or a saturated aqueous solution.

(c) *Sodium Hydroxide Solution (20 g. per l.)*.—Dissolve 20 g. of  $\text{NaOH}$  in ammonia-free distilled water and dilute to 1 liter.

(d) *Nessler's Reagent*.—Dissolve 50 g. of  $\text{KI}$  in the smallest amount of cold distilled water. Add saturated  $\text{HgCl}_2$  solution until an excess is indicated by the first slight formation of a permanent precipitate. Add 400 ml. of  $\text{KOH}$  (550 g. per l.), at which time a heavy white precipitate will form. Add more saturated  $\text{HgCl}_2$  solution until a pale yellow color develops. Dilute to about 1 liter with distilled water and allow to settle. Draw off the clear supernatant liquid for use as Nessler's reagent. The Nessler's reagent should give a distinct coloration with 500 ml. of water containing 0.005 mg. of  $\text{NH}_3$ . If this is not obtained, the reagent can be made more sensitive by the addition of more  $\text{HgCl}_2$ .

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

(e) *Standard Ammonia Solution* (1 ml. = 0.01 mg.  $\text{NH}_3$ ).—Dissolve 3.141 g. of c.p.  $\text{NH}_4\text{Cl}$ , which has been well dried at 100 C., in 1 liter of ammonia-free distilled water in a 1-liter volumetric flask. Pipette 10 ml. of the solution into a 1-liter volumetric flask and dilute to 1 liter with ammonia-free distilled water. This standard is stable and, if kept in a well-stoppered flask, will keep indefinitely.

(f) *Color Standards*.—Pipette into Nessler tubes known volumes of the standard ammonia solution, such that the  $\text{NH}_3$  content increases in 0.01-mg. steps. Dilute each color standard to 50 ml., and then add to each standard 2 ml. of Nessler's reagent (Note).

NOTE: *Stability of Standards*.—Since the color developed changes slowly and continuously, it is important to add the Nessler's reagent to sample and standards at about the same time. The maximum interval for best results is 30 min.

### Preparation of Sample

4. Reduce the sample, if necessary, with a rasp or other means so as to pass a No. 60 (250-micron) sieve, taking care not to overheat the sample. Keep the sample in a tightly stoppered flask until analyzed. Due to the high volatility of ammonia, carry out the analysis as rapidly as possible after reducing the sample.

### Procedure

5. (a) Weigh 10 g. of the prepared sample on the balance to the nearest 0.01 g. Place in the 250-ml. glass-stoppered flask and cover with 100 ml. of ammonia-free distilled water at 90 to 100 C. Stopper and allow to cool at room temperature for 1 hr., while shaking frequently.

(b) Filter rapidly through a medium-porosity sintered-glass filter crucible. Pipette 10 ml. of the filtrate into a

100-ml. Claissen-type distillation flask. Add 1 g. of solid  $\text{KMnO}_4$  or 1.5 ml. of saturated  $\text{KMnO}_4$  solution. Add 10 ml. of  $\text{NaOH}$  (20 g. per l.). Drop a few chips of porous plate into the flask to minimize "bumping." Stopper the flask with rubber stoppers. Mix well, and if necessary, add more  $\text{KMnO}_4$  so that the purple color is maintained. (Note.)

NOTE.—Before any samples are determined, it is advisable to eliminate all traces of  $\text{NH}_3$  from the apparatus by running a blank determination using 10 ml. of ammonia-free distilled water. The apparatus is then ready for a sample determination.

(c) Distill the sample slowly so as to prevent the entrainment of any  $\text{KMnO}_4$ , and collect the first 15 ml. of the distillate in the 50-ml. Nessler tube. Dilute the distillate to the 50-ml. mark with ammonia-free distilled water, and add 2 ml. of Nessler's reagent.

(d) Using the Nesslerimeter, compare the color developed in the Nessler tube containing the sample distillate against the prepared color standards (see Section 3 (f)). For good color definition, the highest concentration of the standard solution used should not exceed 0.06 mg. of  $\text{NH}_3$ . If the color developed in the sample is darker than this, a 10-ml. aliquot of the filtrate may be diluted to a known volume in a volumetric flask and a 10-ml. portion of this diluted solution may be used in the distillation (Paragraph (c)).

### Calculation

6. Calculate the percentage of ammonia in the sample as follows:

$$\text{NH}_3, \text{ per cent} = \frac{SD}{10}$$

where:

$S$  = milligrams of  $\text{NH}_3$  in the matching color standard, and

$D$  = dilution factor (if sample is diluted before distillation).

# *Tentative Method for*

## ESTIMATING BLOCKING OF PLASTIC SHEETS<sup>1</sup>



A.S.T.M. Designation: D 884 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method is intended to give information regarding the degree of blocking of plastic sheets under standard conditions of temperature and pressure. Depending on the end use of the sheet materials, different degrees of blocking may be permissible. No single set of conditions of test can give results indicating the degree of blocking to be expected under all conditions of use (temperature, humidity, pressure, time). Several conditions of test are given to duplicate various conditions of use or to give short-time indications of behavior in use. This method is applicable to comparatively thin, flexible plastic sheets which should show no blocking, or only a slight degree of blocking, during storage and use.

### Blocking

2. Blocking is the adhesion between touching layers of a material, such as

occurs under moderate pressures during storage or use.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Glass Plate*.—Pieces of plate glass 3 by 4 in. by approximately  $\frac{1}{16}$  in. in thickness.

(b) *Paper*.—3 by 5-in. pieces of newsprint approximately 0.003 in. in thickness.

(c) *Oven*.—Constant temperature oven which can be kept at any temperature up to 85 C. within limits of plus or minus 1 C.

(d) *Weights*.—Weights of 4 lb. and 12 lb., having a base area between 2 by 3 in. and 3 by 4 in.

### Test Specimens

4. Twelve test specimens 3 by 5 in. shall be cut from each sample for each test.

### Conditioning Test Specimens

5. (a) Specimens and paper for test at 50 C. (122 F.) or higher, as provided

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

in Section 6 (e), shall be conditioned for 24 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and at  $50 \pm 2$  per cent relative humidity.

(b) Specimens for test at 38 C. (100 F.), as provided in Section 6 (e), shall be conditioned for 24 hr. at  $25 \pm 1$  C. ( $77 \pm 2$  F.), and in a saturated atmosphere (Note).

NOTE.—Saturated atmosphere is defined as the conditions existing in a space where the capacity to hold moisture as a pure gas has been exceeded, producing condensation, precipitation, dew, etc. This condition is sometimes erroneously referred to as 100 per cent relative humidity.

### Procedure

6. Many plastic sheets are of such composition that their two surfaces (back and face) show different degrees of blocking. The three possible surface combinations (face to face, face to back, and back to back) shall be tested under a pressure of  $\frac{1}{3}$  or 1 psi, as follows:

(a) Make a pile-up consisting of three sandwich combinations, the first being prepared as follows:

3 by 4 in. glass plate,  
3 by 5 in. sheet of newsprint,  
3 by 5 in. specimen, face up,  
3 by 5 in. specimen, face down,  
3 by 5 in. sheet of newsprint, and  
3 by 4 in. glass plate.

Repeat this combination of papers (Note), specimens and glass plates except that in the second sandwich place the specimens back to back. Repeat a third time but with the specimens back to face.

NOTE.—Paper may be omitted if there is no danger of the specimen sticking to the glass plates. When specimens are to be tested at 38 C. (100 F.), as provided in Paragraph (e), use a thin, well-calendered paper and condition it in the same manner as the specimens.

(b) Align specimens, paper, and glass plates so that they are flush along one 3-in. edge and so that specimens and

paper extend 1 in. beyond the glass plates along the other 3-in. edge.

(c) Place the weight on top of the pile-up, using the 4-lb. weight for a pressure of  $\frac{1}{3}$  psi., and the 12-lb. weight for a pressure of 1 psi.

(d) For a duplicate test make a second pile-up as described in Paragraphs (a) to (c).

(e) Place the pile-ups with weights thereon in an oven for 24 hr. maintained at one of the following test temperatures as specified:

$50 \pm 1$  C. ( $122 \pm 2$  F.), or  
 $60 \pm 1$  C. ( $140 \pm 2$  F.), or  
 $70 \pm 1$  C. ( $158 \pm 2$  F.), or  
 $82 \pm 1$  C. ( $180 \pm 2$  F.), or  
 $38 \pm 1$  C. ( $100 \pm 2$  F.), saturated atmosphere  
(See Note, Section 5).

(f) Remove the pile-ups from the oven and keep them at room temperature for at least 2 hr. but not longer than 24 hr.

(g) Remove the successive layers and note the degree of blocking between the three surface combinations which shall be judged as follows:

*Degree N.B.*—No blocking. No adhesion between sheets; sheets slide freely.

*Degree V.Sl.B.*—Very slight blocking. Sheets do not slide freely, but can be made to slide by application of slight force.

*Degree Sl.B.*—Slight blocking. Sheets have to be peeled apart, but surfaces remain intact.

*Degree B.*—Blocking. Sheets have to be peeled apart and surfaces are damaged.

### Report

7. The report shall include the following:

- (1) Identification of material,
- (2) Degree of blocking for each

combination of surfaces, for example, *ff-Sl.B.*, *bb-B.* (that is, face to face, slight blocking; back to back, blocking),

(3) Pressure applied ( $\frac{1}{3}$  or 1 psi.), and

(4) Time and temperature of exposure. When the specimens have been conditioned at 38 C. (100 F.), as provided in Section 6 (e), report also how the nominal "saturated atmosphere" was obtained.



# *Tentative Method of Test for*

## BRITTLE TEMPERATURE OF PLASTICS AND ELASTOMERS<sup>1</sup>



A.S.T.M. Designation: D 746 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test establishes a temperature above which compositions of plastics and elastomers are not brittle when subjected to the conditions herein specified.

NOTE.—This test does not take into account time effects such as may be induced by low temperature incompatibility of plasticizers in the material under test.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Specimen Clamp and Striking Member*.—The specimen clamp, designed to hold the specimen as a cantilever beam, and the striking edge shall be capable of motion relative to each other at a constant linear speed of  $6.5 \pm 0.5$  ft. per sec. at and during impact. The distance between the center line of the striking edge and the clamp shall be  $0.25 \pm 0.01$  in. at impact. The striking edge shall have a radius of  $0.062 \pm 0.005$  in. These dimensional requirements for the

recommended apparatus are shown in Fig. 1.

NOTE.—A recommended apparatus is shown in Fig. 2. The striking arm is driven by a motor which may require as many as 8 revolutions in order to attain its steady-state speed. A stationary specimen is clamped in a yoke which is centered about the drive shaft of the striking arm and is locked in the position shown during conditioning and impact. When unlocked, the yoke may be rotated out of the tank for inspection of the specimen just tested and insertion of the new specimen. The worm and gear disengage after impact and a catch (not shown) is provided to prevent the striking arm from falling back against the specimen.

(b) *Thermometer*.—A thermometer graduated in 1 C. divisions and having a range suitable for the temperatures at which determinations are to be made shall be used. It shall be located as near the specimen as possible.

(c) *Heat-Transfer Medium*.—Any heat-transfer medium which remains fluid at the test temperatures and which will not appreciably affect the materials being tested may be used. Among the liquids that may be found suitable for low-temperature use are acetone, ethyl alcohol, butyl alcohol, normal hexane, and methyl phosphate and water in the

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

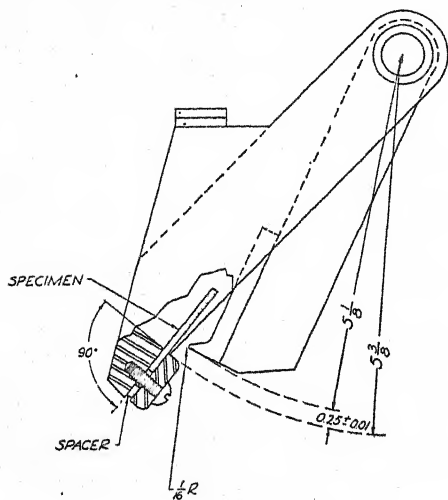
ratio of 87 to 13 by volume. Acetone, methyl alcohol, and ethyl alcohol are suggested for use with natural and synthetic rubbers. For temperatures down to  $-120^{\circ}\text{C}$ . methylcyclohexane and liquid nitrogen may be suitable. If the brittle temperature of specimens immersed for 1 hr. in the heat-transfer medium in the vicinity of their brittle temperature differ appreciably from others soaked for 3 min. and tested at the same temperature, the heat-transfer me-

2) or a test chamber (for air) shall be provided as a part of the test apparatus.

(f) *Stirrer*.—A stirrer for liquids or a fan or blower for air, which insures thorough circulation of the heat transfer medium, shall be provided.

### Test Specimens

3. The test specimens shall be  $1.50 \pm 0.05$  in. in length,  $0.250 \pm 0.015$  in. in width, and  $0.075 \pm 0.010$  in. in thickness. Other values of specimen thickness may be used provided it can be shown that they give equivalent results for the material being tested.



All dimensions in inches.

FIG. 1.—Dimensional Requirements for Specimen Clamp and Striking Arm.

dium shall be considered to affect the material. In any case, air shall be the reference heat-transfer fluid or medium.

(d) *Temperature Control*.—Suitable means (automatic or manual) shall be provided for controlling the temperature of the heat-transfer medium within plus or minus  $0.5^{\circ}\text{C}$ . of the desired value. For liquids, powdered dry ice (solid  $\text{CO}_2$ ) and an electric immersion heater are recommended means of accomplishing this.

(e) *Tank or Test Chamber*.—A tank (for liquid heat-transfer media, see Fig.

### Procedure with Liquid Heat Transfer Medium

4. (a) Before running a test, the bath shall be prepared and the apparatus brought to the lowest temperature to be used. This may be accomplished by placing approximately 4 lb. of powdered dry ice in the insulated tank and slowly adding the heat-transfer liquid until the tank is filled to a level approximately 1 to 2 in. from the top. During the test the temperature of the bath may be maintained constant by the judicious addition of small quantities of powdered dry ice. Change of temperature will require either the addition of more dry ice or use of the electric immersion heater.

(b) The test specimen shall be mounted in the apparatus and submerged for  $2.5 \pm 0.5$  min. at the test temperature. In the case of the recommended apparatus this shall be done as follows: Rotate the specimen clamp yoke out of the bath and insert a test specimen. Tighten the specimen clamp slightly, lower the yoke into the bath, and lock the specimen yoke firmly in the testing position. Adjust the striking arm to the position shown in Fig. 2.

(c) After immersion for  $2.5 \pm 0.5$  min. at the test temperature, the tem-

perature shall be recorded and the impact blow delivered.

(d) The specimen clamp shall be removed from the bath and the specimen examined to determine whether or not it has failed. Failure is defined as the division of the test specimen into two or more completely separated pieces. The

(f) In establishing the lowest temperature of nonfailure of a material, it is recommended that the test be started at  $-70^{\circ}\text{C}$ . or at a temperature below the expected temperature of nonfailure and specimens tested at intervals of  $10^{\circ}\text{C}$ ., one at each temperature, until a failure is not obtained. The bath temperature

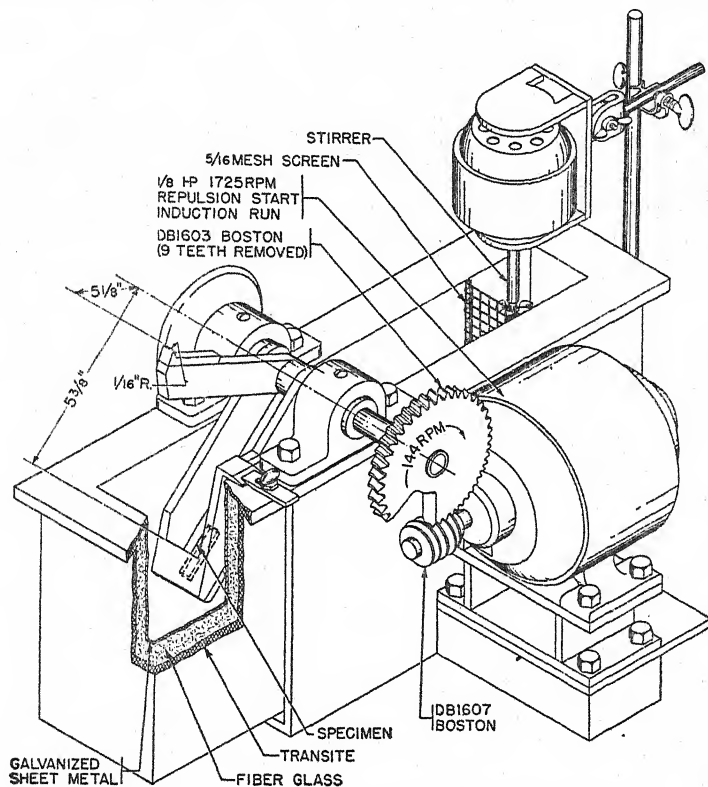


FIG. 2.—Apparatus for Brittle Temperature Test of Plastics.

occurrence of fissures or cracks does not constitute brittle-type failure although their presence shall be recorded in the report. For the apparatus shown in Fig. 2 it is important that the striking arm be rotated out of the bath before the specimen. This prevents contact between the striking arm and specimen prior to inspection.

(e) A new specimen shall be used for each test or impact.

shall then be decreased to the highest value at which a failure was observed and tests made at successively increasing temperatures. Steps of  $2^{\circ}\text{C}$ . are recommended. Tests shall be made at each temperature until five consecutive cases of nonfailure at one temperature are observed, or until one failure is observed. If failure occurs, proceed to the next higher temperature. The lowest temperature of nonfailure of five consecutive

specimens shall be recorded as the brittle temperature of the test materials.

#### Procedure With Air as the Heat Transfer Medium

5. (a) Before running a test, the refrigerating unit shall be adjusted, and the test chamber, test apparatus, and specimens shall be brought to thermal equilibrium at the desired temperature.

(b) The test chamber shall be opened and the specimen tested in accordance with the procedure described in Sections 4 (b) to (f), except that the specimen shall be inserted in the clamp using forceps to avoid warming the specimen with the hands, and after closing the chamber the

impact blow shall not be initiated until 5 min. after the air in the test chamber has returned to its equilibrium value.

#### Report

6. The report shall include the following:

(1) The brittle temperature, which is defined as the lowest temperature of non-failure of five consecutive specimens under the specified test conditions,

(2) Complete identification of the material tested, including type, source, manufacturer's code designation, form, and previous history,

(3) Thickness of test specimen,

(4) Heat transfer medium used, and

(5) Date of test.

# *Tentative Method of Test for*

## COLORFASTNESS OF PLASTICS TO LIGHT<sup>1</sup>



A.S.T.M. Designation: D 620 - 45 T

ISSUED, 1941; REVISED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method of test is intended to establish a laboratory procedure for evaluating the relative resistance of plastics to change in color when exposed to sunlight (Note). Plastics may change in color because of the action of solar radiation on the dyes or pigments or on the plastic base.

NOTE.—Some correlation has been observed between color changes in plastics resulting from exposure to the conditions of this test method and those resulting from a much longer exposure to sunlight, but no general assumption as to the existence or the extent of such correlation should be made.

(b) This method is not intended to show the resistance of plastics to weathering, that is, continuous exposure of the materials out-of-doors.

### Apparatus

2. The apparatus (Fig. 1) shall consist of the following:

(a) *Lamp*.—A General Electric sun-lamp, model BM 12, or an equivalent

lamp, equipped with a reflector approximately 15 in. in diameter at the lower rim, and an S-1 bulb which has been in use at least 50 hr. and less than 550 hr. The S-1 bulb consists of a combination tungsten filament - mercury arc enclosed in Corex D glass which absorbs most of the ultraviolet radiation below 2800 Å (Note 1). The bulb is rated at 400 w. The operating voltage shall be maintained at  $110 \pm 2$  v. (Note 2).

NOTE 1.—To measure the ultraviolet light output of the S-1 bulb, the actinometer cell using an oxalic acid - uranyl sulfate solution may be used as described in the Tentative Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (A.S.T.M. Designation: D 749).<sup>3</sup> As no minimum value for the ultraviolet output has been established, actinometer data are useful only for comparisons between lamps.

NOTE 2.—To control voltage within the limits specified, an automatic voltage-stabilizer such as General Electric stabilizer, Catalogue No. 68G158, may be used.

(b) *Disk*.—A phonograph turntable, operating at 33 rpm., on which shall be mounted a light-colored corrosion-re-

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, May 10, 1945.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

sistant metal disk approximately 17 in.<sup>4</sup> in diameter and 0.1 in. in thickness. Two sets of 21 brass machine screws and nuts shall be attached to the disk at holes equally spaced on concentric circles of approximately  $3\frac{3}{4}$ -in. and 6-in. radius, respectively. The screws may be size No. 10, 32 threads to the inch,  $\frac{1}{2}$  in. in length with fillister head. Brass washers about  $\frac{5}{8}$  in. in diameter and 0.05 in. in

mately one third of the outer portion of the specimen.

### Test Specimen

3. The test specimen shall be approximately 2 in. in length and 1 in. in width. The material shall be tested in the thickness as received.

NOTE.—It is desirable to include a test specimen of known discoloration behavior in each

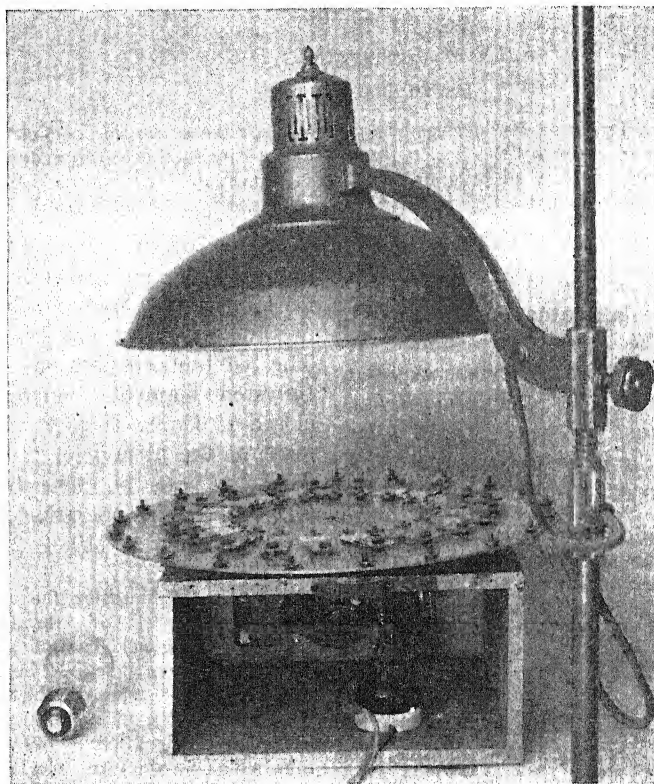


FIG. 1.—Apparatus for Colorfastness Test of Plastics.

thickness shall be placed over the nuts to support the specimens about  $\frac{3}{16}$  in. above the surface of the disk. A brass plate about 1 in. square and 0.05 in. in thickness shall be fitted over each of the screws of the outer row to cover approxi-

test series in order to guard against the use of a defective bulb. A specimen of transparent cellulose nitrate plastic  $\frac{1}{8}$  in. in thickness has been found to be satisfactory for this purpose.

### Procedure

4. The specimens shall be placed on the disk so that they are supported above its surface by the washers. The disk

<sup>4</sup>This equipment is used extensively in accelerated weathering tests specified by several government agencies. These tests require the 17-in. diameter disk and the outer row of screws shown in Fig. 1.

shall be centered under the S-1 bulb so that the bottom plane of the specimens is 7 in. from the bottom of the bulb. The test shall be so conducted that an A.S.T.M. Partial Immersion Thermometer 1C - 39 as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1),<sup>3</sup> placed on the turntable with its midpoint at the center of the disk, will read 55 to 60 C. (Note 1). If the thermometer reads higher than 60 C. a fan shall be provided, placed so that it blows against the bottom of the turntable a volume of air sufficient to bring the thermometer reading to 55 to 60 C. (Note 2).

NOTE 1.—The thermometer does not necessarily indicate the temperature of the samples, since the reading of the thermometer is determined partly by the amount of radiant energy which it absorbs and partly by the temperature of its surroundings.

NOTE 2.—If the apparatus is not enclosed, and operates in a room in which the ambient temperature is 70 to 80 F., the thermometer reading will lie between 55 and 60 C. If the reading is below 55 C., the temperature can be raised by reducing somewhat the air circulation around the lamp.

### Report

5. (a) The relative amount of color change of the plastic shall be reported as none, slight, appreciable, or extreme.

NOTE.—A slight change is defined here as one which is perceptible with difficulty. An appreciable change is one which is readily perceptible without close examination but is insufficient to markedly alter the original color of the specimen. An extreme change is one which is very obvious and has resulted in a marked alteration of the original color of the specimen.

(b) The character of the color change, for example, lighter or darker, and any other noteworthy effect of the exposure to the light on the appearance or condition of the specimen shall be reported.



# *Tentative Method of Test for*

## COMPRESSIVE STRENGTH OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 695 - 44 T

ISSUED, 1942; REVISED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method is intended for use in determining the comparative compressive properties of organic plastics in the form of test specimens of standard shape and when tested under defined conditions of pretreatment, temperature, humidity, and testing machine speed. (Notes 1, 2, 3, and 4.)

### Definition of Terms<sup>3</sup>

2. (a) *Compressive Stress* (nominal) is the compressive load per unit area of original cross-section carried by the test specimen at any time during a compression test. It is expressed in pounds per square inch.

(b) *Compression* is the change in length produced in a longitudinal section of the test specimen by a compressive load. It is expressed in inches.

(c) *Compressive Strain* (or *Unit Compression*) is the ratio of the compression to the original length of the measured compressing section of the test specimen,

that is, the change in length per unit of original length. It is expressed as a dimensionless ratio.

(d) *Percentage Compression* is the compression at any time expressed as a percentage of the original length of the measured compressing section. It is equal to one hundred times the compressive strain measured at the time in question.

(e) *Compressive Yield Stress* is the compressive stress (nominal) at the first observable point in a compression test where compression of the specimen is increasing but compressive stress (nominal) is remaining constant. It is expressed in pounds per square inch.

(f) *Percentage Compression at Compressive Yield Stress* is the compression of the test specimen at the first observable point in a compression test where compression of the test specimen is increasing but compressive stress (nominal) is remaining constant, expressed as a percentage of the original length of the measured compressing section.

(g) *Offset Yield Stress* is the stress at which the stress-strain curve departs from linearity by a specified percentage of deformation (offset). The method

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, January 1, 1944.

<sup>3</sup> Attention is also directed to the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6), see p. 487.

for determining the offset yield stress is prescribed in the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6)<sup>4</sup> under definition of yield strength, Paragraph 2 (a) on "Offset Method." The amount of offset shall always be stated, for example, 0.2 per cent offset yield stress.

(h) *Compressive Strength* is the maximum compressive stress (nominal) carried by a test specimen during a compression test. It may or may not be the compressive stress (nominal) carried by the specimen at the moment of rupture. (Note 5.) It is expressed in pounds per square inch.

(i) *Percentage Compression at Failure* is the compression of the test specimen at the moment of rupture expressed as a percentage of the original length of the measured compression section. (Note 6.)

(j) *Compressive Stress-Strain Curve* is the curve obtained by plotting compressive stresses (nominal) as ordinates against corresponding compressive strains as abscissas for the entire course of a compression test.

(k) *Elastic Modulus* is the ratio within the elastic limit of compressive stress (nominal) to corresponding compressive strain. (Note 7.) It is expressed in pounds per square inch.

(l) *Mean Rate of Stressing* is, for report purposes, the compressive stress (nominal) in pounds per square inch carried by the test specimen at the moment when the compressive strain is 0.02, or at the moment of rupture if this occurs at a compressive strain below 0.02, divided by the time in seconds required for the attainment of this stress and strain, measured from the beginning of application of the load. It is expressed in pounds per square inch per second. (Note 8.)

(m) *Mean Rate of Straining* is, for report purposes, the mean rate of compression of the test specimen divided by the original length of the compressing section. Under the defined conditions of test it is essentially equal to the mean rate of crosshead movement, in inches per second, divided by the original length of the test specimen, in inches, and is expressed as units per second. (Note 9.)

### Apparatus

3. The apparatus shall consist of the following:

(a) *Testing Machine*.—Any suitable testing machine of the constant-rate-of-crosshead-movement type and comprising essentially the following:

(1) *Drive Mechanism*.—A drive mechanism for imparting to the crosshead a uniform, controlled velocity with respect to the base, this velocity to be regulated as specified in Section 7.

(2) *Load Indicator*.—A load-indicating mechanism capable of showing the total compressive load carried by the test specimen. This mechanism shall be essentially free from inertia-lag at the specified rate of testing and shall indicate the load with an accuracy of plus or minus 1 per cent of the indicated value, or better. The accuracy of the testing machine shall be verified in accordance with the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials<sup>4</sup> (Note 10.)

(3) The testing machine shall be constructed of such material and in such proportions that the total elastic longitudinal strain of the system does not exceed 1 per cent of the total compressive strain of the test specimen at any time during the test and at any load up to the rated capacity of the machine.

(b) *Compression Tool*.—A compression tool for applying the load to the test specimen. This tool shall be so con-

<sup>4</sup>Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

structed that loading is truly axial, and so that the load is applied through surfaces which are flat and parallel to the ends of the specimen and are normal to the long axis of the specimen. A suitable form of compression tool is shown in Fig. 1 and consists of the following:

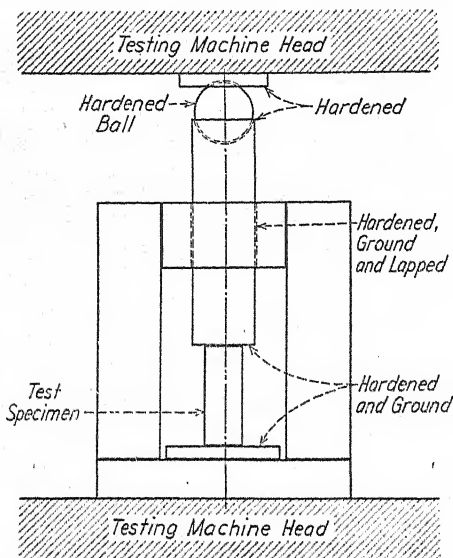


FIG. 1.—Compression Tool.

(1) *Base Plate*.—A hardened steel base plate, the upper surface of which is machined and polished to a flatness as nearly perfect as possible.

(2) *Plunger and Ball*.—A hardened steel plunger, the lower end of which is machined and polished to a flatness as nearly perfect as possible and the upper end of which is machined and polished to a hollow hemisphere to receive a polished, hardened steel ball, the diameter of which approaches that of the plunger.

(3) *Guiding Framework*.—A guiding framework attached to the base plate and holding the plunger so that its longitudinal axis is perpendicular to the surface of the base plate and so that its polished lower surface is parallel to the

polished surface of the base plate within 0.001 in. over the entire contact area.

(4) *Bushing*.—A bushing holding the plunger in the guiding framework. It shall be smooth and provided with adequate lubrication so that negligible force is required to move the plunger freely through the bushing.

(5) In use, the base of this compression tool shall be centered on the lower platen of the testing machine and contact with the top platen (or crosshead) shall be made between the hardened steel ball and a hardened steel plate attached to the top platen. (Note 11.)

(c) *Extension Indicator*.—A suitable instrument for determining the distance between the contact surfaces of the compression tool (that is, the length of the test specimen) or the distance between two fixed points on the test specimen at any time during the test. It is desirable, but not essential, that this instrument automatically record this distance (or any change in it) as a function of the load on the test specimen or of the elapsed time from the start of the test, or both. This instrument shall be essentially free of inertia-lag at the specified rate of loading, and shall be accurate to plus or minus 1 per cent or better.

(d) *Micrometers*.—Suitable micrometers, reading to at least  $0.001 \pm 0.000$  in., for measuring the width, thickness, and length of the test specimens.

(e) *Oven and Desiccators*.—A circulating air oven, adjustable to  $50 \pm 3$  C. ( $122 \pm 5$  F.), and desiccators containing anhydrous calcium chloride (or other suitable desiccant) are required for conditioning some materials.

#### Test Specimens

4. (a) The test specimen shall be in the form of a right cylinder or prism whose length is twice its principal width or diameter, and whose ends are parallel

within 0.0001 in. A prism, of square cross-section, 0.5 by 0.5 by 1.0 in., shall be considered the standard test specimen (Note 12.) shape and shall be used for comparative purposes whenever possible. Where only elastic modulus and offset yield stress data are desired, the test specimen shall be of such dimensions that the slenderness ratio (length divided by the least radius of gyration) shall be in the range of 11 to 15. For specimens of rectangular cross-section, the radius of gyration shall be 0.289 times the smaller cross-sectional dimensions. For specimens of circular cross-section, the radius of gyration shall be 0.250 times the diameter.

(b) The specimen may be prepared by machining operations from materials in sheet, plate, slab, rod, tube, or similar form, or it may be prepared by compression molding or injection molding of the material to be tested. The standard prism, 0.5 by 0.5 by 1.0 in., may be cut from bars 0.5 by 0.5 by 5.0 in., which are standard for a number of other tests, for example, flexural strength, distortion temperature, impact strength, etc. (Note 13.)

(c) Where it is desired to test materials in the form of sheets the thickness of which is substantially less than 1.0 in., a pile-up of sheets 0.5 in. square with a minimum number of layers to produce a height of at least 1.0 in. may be used. (Note 14.)

(d) When testing materials that may be suspected of anisotropy, duplicate sets of test specimens shall be prepared having their long axes respectively parallel with and normal to the suspected direction of anisotropy. (Note 15.)

(e) All machining operations shall be done carefully, so that smooth surfaces result. Great care shall be taken in machining the ends so that smooth parallel surfaces, perpendicular to the long axes of the specimen, result.

## Conditioning

5. (a) Plastic materials may be preconditioned for testing under either "dry" or "standard" atmospheric conditions. (Note 16.) In general, the preconditioning method is chosen on the basis of the rapidity with which the compressive properties of the material in question vary with changes in atmospheric humidity and temperature. Thus, it is known that the compressive properties of cellulose derivatives and of some polyvinyl acetal resins change with comparative rapidity when subjected to rapidly changing temperature or humidity, whereas the compressive properties of polystyrene, polymethacrylic acid esters, phenolformaldehyde, and urea-formaldehyde plastics are only slowly affected by such atmospheric changes. Hence, the latter group of materials has most frequently been prepared for testing by "dry" preconditioning methods, whereas the former group has most frequently been prepared by "standard" preconditioning methods.

(b) *"Standard" Conditioning Procedures.*—Specimens shall be preconditioned for at least 14 days in an enclosure maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity prior to testing. The actual tests shall be conducted at the same atmospheric conditions. (Note 17.)

(c) *"Dry" Conditioning Procedures.*—Specimens shall be preconditioned for a period of 48 hr. in a circulating air oven at a temperature of  $50 \pm 3$  C. ( $122 \pm 5$  F.) They shall then be removed from the oven, cooled in a desiccator over anhydrous calcium chloride (or an equivalent desiccant) for a period of at least 16 hr. prior to testing. The test shall be conducted in an enclosure maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity. A definite time interval, previously decided upon by co-operating laboratories, shall be allowed to

elapse between removal of the specimens from the desiccator and the application of load in the test.

(d) Special conditioning procedures may be used by agreement between co-operating laboratories.

(e) If no conditioning method is specified, the "standard" conditioning procedure shall be used. (Note 18.)

#### Number of Test Specimens

6. (a)\* At least five specimens shall be tested for each sample in the case of isotropic materials.

(b) Ten specimens, five normal to, and five parallel with the principal axis of anisotropy, shall be tested for each sample in the case of anisotropic materials.

(c) Specimens that break at some obvious fortuitous flaw shall be discarded and retests made, unless such flaws constitute a variable the effect of which it is desired to study.

(d) Results that deviate from the mean value of all tests should be rejected if the deviation of the doubtful value is more than five times the average deviation from the mean obtained by excluding the doubtful value. Such doubtful values shall be discarded and retests made, unless the degree of variability is a factor that is being studied.

#### Speed of Testing

7. (a) Speed of testing is defined as the relative velocity of approach of the two crossheads of the testing machine when running idle (under no load).

(b) The standard speed of testing shall be 0.20 to 0.25 in. per min.

(c) If load-deformation data are being taken, as for determination of elastic modulus or for study of the initial portion of the stress-strain curve, the speed of testing shall be 0.05 in. per min. until the "yield point" is reached. The rate of crosshead movement shall then be in-

creased to the standard speed of 0.20 to 0.25 in. per min., and maintained at that speed until failure of the specimen. (Note 19.)

#### Procedure

8. (a) The actual testing shall be carried out in an atmosphere maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity.

(b) The width and thickness of the specimen shall be measured to the nearest 0.001 in., at several points along its length. The minimum values of area shall be recorded. The length of the specimen shall be measured to the nearest 0.001 in. and the value recorded.

(c) The test specimen shall be placed between the surfaces of the compression tool, taking care to align the center line of its long axis with the center line of the plunger and to insure that the ends of the specimen are parallel with the surfaces of the compression tool. (Note 20.) The plunger shall be brought down against the top of the specimen and the crosshead of the testing machine adjusted until it just contacts the ball on top of the plunger.

(d) If stress-strain data are desired, the procedure shall be as follows:

(1) Attach the extension recorder.

(2) Set the speed control at 0.05 in. per min. and start the machine.

(3) Record loads and corresponding compressions at appropriate intervals of strain. (Note 21.)

(4) Record the load carried by the specimen when the compressive strain reaches 0.02 and the elapsed time from the start of the test until this point is reached. If rupture occurs before the compressive strain reaches 0.02, record the elapsed time from the start of the test until the specimen breaks. (Note 21.)

(5) Record the load carried by the

specimen and the compression at the yield point. (Note 21.)

(6) After the yield point has been reached (or after the strain has passed 0.02), increase the speed to 0.20 to 0.25 in. per min. and allow the machine to run at this speed until the specimen breaks.

(7) Record the maximum load carried by the specimen during the test (usually this will be the load at the moment of rupture). (Note 21.)

(8) Record the compression at the moment of rupture of the specimen.

(e) If only compressive strength and percentage compression data are desired, the procedure shall be as follows:

(1) Set the speed control at 0.20 to 0.25 in. per min. and start the machine.

(2) Record the load carried by the specimen when the compressive strain reaches 0.02 and the elapsed time from the start of the test until this point is reached. If rupture occurs before the compressive strain reaches 0.02, record the elapsed time from the start of the test until the specimen breaks. (Note 21.)

(3) Record the load carried by the specimen and the compression at the yield point. (Note 21.)

(4) Record the maximum load carried by the specimen during the test (usually this will be the load at the moment of rupture). (Note 21.)

(5) Record the compression at the moment of rupture of the specimen.

### Calculations

9. (a) *Compressive Strength* shall be calculated by dividing the maximum compressive load carried by the specimen during the test by the original cross-sectional area of the specimen. The result shall be expressed in pounds per square inch and reported to three significant figures.

(b) *Percentage Compression at Failure* shall be calculated by dividing the compression of the specimen at the moment

of rupture by the original length of the measured compressing section of the test specimen and multiplying by one hundred. The percentage compression shall be reported to two significant figures.

(c) *Compressive Yield Stress* shall be calculated by dividing the load carried by the specimen at the first observable point in the test where the compression of the specimen is increasing, without increase in the load carried by the specimen, by the original cross-sectional area of the specimen. The result shall be expressed in pounds per square inch and reported to three significant figures.

(d) *Percentage Compression at Yield* shall be calculated by dividing the compression of the specimen at the first observable point in the test where the compression is increasing, without increase in the load carried by the specimen, by the original length of the measured compressing section of the test specimen and multiplying by one hundred. The percentage compression shall be reported to two significant figures.

(e) *Offset Yield Stress* shall be calculated by the method referred to in Section 2 (g).

(f) *Elastic Modulus* shall be calculated by drawing a tangent to the initial linear portion of the stress-strain curve, selecting any point on this straight line and dividing the compressive stress (nominal) represented by the point by the corresponding compressive strain. The result shall be expressed in pounds per square inch and reported to three significant figures.

(g) *Mean Rate of Stressing* shall be calculated by dividing the compressive load carried by the specimen when the strain reaches 0.02 or at the moment of rupture, whichever occurs first, by the original minimum cross-sectional area of the specimen, and then dividing this result by the time in seconds, measured



from the beginning of the test, required to attain this compressive load and strain. The result shall be expressed in pounds per square inch per second and reported to three significant figures.

(h) *Mean Rate of Straining* shall be calculated by dividing the mean rate of compression of the test specimen by the original length of the specimen. The result shall be expressed as a dimensionless ratio per second and reported to three significant figures. (Note 22.)

(i) For each series of tests, the arithmetic mean of all values obtained shall be calculated to three significant figures and reported as the "average value" for the particular property in question.

(j) The deviation of each value from the "average value" shall be calculated and the arithmetic mean of these deviations determined. This arithmetic mean shall be reported to two significant figures as the "average deviation" of the particular series of results. Retain two significant figures.

### Report

10. The report shall include the following:

(1) Complete identification of the

material tested, including type, source, manufacturer's code numbers, form, principal dimensions, previous history, etc.,

(2) Method of preparing test specimens,

(3) Type of test specimen and dimensions,

(4) Conditioning procedure used,

(5) Atmospheric conditions in test room,

(6) Number of specimens tested,

(7) Speed of testing,

(8) Mean rate of stressing as calculated in Section 9 (f),

(9) Mean rate of straining as calculated in Section 9 (g),

(10) Compressive strength, average value and average deviation,

(11) Percentage compression at failure, average value and average deviation.

(12) Compressive yield stress, average value and average deviation,

(13) Percentage compression at yield, average value and average deviation.

(14) Elastic modulus in compression, average value and average deviation, and

(15) Date of test.

### EXPLANATORY NOTES

NOTE 1.—This method is not well adapted to the testing of plastics in the form of thin sheets or films. Neither is this method particularly suitable for the testing of soft rubber or rubber-like polymers (elastomers), although the position of such materials as organic plastics is recognized.

NOTE 2.—This method may be used for testing phenolic resin molded or laminated materials. However, where correlation with previous data is an object, such materials should be tested in accordance with the following methods of the American Society for Testing Materials:

Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48),<sup>4</sup>

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229),<sup>4</sup>

Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (A.S.T.M. Designation: D 348),<sup>4</sup>

Standard Methods of Testing Laminated Round Rods Used in Electrical Insulation (A.S.T.M. Designation: D 349),<sup>4</sup> and

Tentative Method of Test for Compressive Strength of Electrical Insulating Materials (A.S.T.M. Designation: D 649).<sup>4</sup>

NOTE 3.—It is realized that a material cannot be tested without also testing the method of preparation of that material. Hence, when comparative tests of materials *per se* are desired, the



greatest care must be exercised to insure that all samples are prepared in exactly the same way. Similarly, for referee or comparative tests of any given series of specimens, care must be taken to secure the maximum degree of uniformity in details of preparation, treatment, and handling.

NOTE 4.—This method is not intended to be a precise physical procedure. It is recognized that the constant-rate-of-crosshead-movement type of test leaves much to be desired from a theoretical standpoint, and that the testing speeds disguise important effects characteristic of materials in the plastic state. Further, it is realized that variations in the thicknesses of test specimens, which are permitted by these procedures, produce variations in the surface-volume ratios of such specimens, and that these variations may influence the test results. Hence, where directly comparable results are desired, all specimens should be of equal thickness. Special additional tests should be used where more precise physical data are needed.

NOTE 5.—In the case of a material that fails in compression by a shattering fracture, the compressive strength has a very definite value. In the case of materials that do not fail in compression by a shattering fracture, the value obtained for compressive strength is of arbitrary value depending upon the degree of distortion that is regarded as indicating complete failure of the material. Many plastic materials will continue to deform in compression until a flat disk is produced, the compressive stress (nominal) rising steadily in the process, without any well-defined fracture occurring. Compressive strength can have no real meaning in such cases. It is suggested that the compressive yield strength of such materials be used as a comparative reference point.

NOTE 6.—Where no well-defined rupture of the specimen occurs in compression, no real value for percentage compression at failure can be obtained (see Note 6 above).

NOTE 7.—Since the existence of a true elastic limit in plastics, as in many other organic materials and in many metals, is debatable, the propriety of applying the term "elastic modulus" in its quoted generally accepted definition to describing the "stiffness" or "rigidity" of a plastic has been seriously questioned. The exact stress-strain characteristics of plastic materials are highly dependent on such factors as rate of application of stress, temperature, previous history of specimen, etc. However, stress-strain curves for plastics determined as specified in this method almost always show a linear region at low stresses, and a straight line drawn tangent to this portion of the curve permits

calculation of an elastic modulus of the usually defined type. Such a constant is useful if its arbitrary nature and dependence on time, temperature, and similar factors is realized.

NOTE 8.—It is realized that "mean rate of stressing" as defined in these methods has only limited physical significance. It does, however, roughly describe the average rate at which most of the compressive stress carried by the test specimen is applied and for that portion of the stress-strain curve in which principal stressing occurs. It is affected by the elasticity of the material being tested but is fairly accurately determined by the method described. It can, if desired, be determined more precisely by calculation from load-time data, recorded especially for the purpose during a test. Specification of it is believed to be significant and a valuable adjunct to "rate of straining" calculated from rate of crosshead movement of the testing machine.

NOTE 9.—Mean rate of straining determined in this way is only a first approximation, since no test machine is perfectly rigid. Where more precise data on straining rate are required, they should be determined by calculation from the slope of a strain-time curve, plotted for the purpose, using special data of strain recorded at definite intervals of time during the course of a test.

NOTE 10.—Experience has shown that many testing machines now in use are incapable of maintaining accuracy for as long as the periods between inspection recommended in Standard Methods E 4.<sup>4</sup> Hence, it is recommended that each machine be studied individually and verified as often as may be found necessary. It will frequently be necessary to perform this function daily.

NOTE 11.—Conventional bearing blocks, such as are customarily furnished by manufacturers of testing machines, have been found unsatisfactory for general use, particularly where testing-machine head motion is not truly axial or is variable. While such bearing blocks give good results on hard, brittle materials, or when used with precisely constructed testing machines in first-class condition, the use of a compression tool has been found necessary to insure against buckling of the test specimen when working with the softer plastics.

NOTE 12.—Specimens with the length/thickness ratio of 2.0 as specified are long enough to minimize "end effects" but short enough to avoid trouble with buckling.

NOTE 13.—Molds for the standard test bar, 0.5 by 0.5 by 5.0 in., are described in the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insu-

lation (A.S.T.M. Designation: D 647) of the American Society for Testing Materials.<sup>4</sup>

NOTE 14.—Great care is necessary in testing such pile-ups to avoid buckling.

NOTE 15.—Before testing, all transparent specimens should be given a polariscopic inspection and those which show atypical or concentrated strain patterns should be rejected unless these "initial" strains constitute a variable the effect of which it is desired to measure.

NOTE 16.—In general, the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials<sup>4</sup> will be found useful.

NOTE 17.—In cases of extreme urgency, where the 14-day conditioning period is intolerable, the conditioning may, by agreement between co-operating laboratories, be reduced to a minimum of 48 hr.

NOTE 18.—It is obvious that when exact comparative data are desired, comparable condition-

ing procedures should be employed. It is urged that "standard" conditions be employed for all tests intended for data, specification, or similar purposes, in so far as this is practicable.

NOTE 19.—Such composite tests should not be made when exact data on compressive strength or percentage compression at failure are desired. Such tests should be made at the standard speed of 0.20 to 0.25 in. per min. from beginning to end of the test.

NOTE 20.—Light punch marks at the center of the end of the plunger and at the corresponding point on the base plate will facilitate this adjustment.

NOTE 21.—Loads should be corrected to include the dead weight of those parts of the compression tool which are carried by the specimen.

NOTE 22.—Where the standard specimen 1.0 in. in length is used, mean rate of straining is the same as the rate of crosshead movement in inches per second (for example, a crosshead movement of 0.05 in. per min. is equivalent to a strain of 0.000833 per sec.).

# *Tentative Methods of Test for*

## DEFORMATION OF PLASTICS UNDER LOAD<sup>1</sup>



**A.S.T.M. Designation: D 621 - 45 T**

ISSUED, 1944; REVISED, 1945.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These methods of test cover two procedures for determining the deformation under compression of non-metallic sheet and molded plastic materials, of all classes and all commercial thicknesses, intended for structural and insulating purposes.

(b) The word deformation is used herein in the broad sense to cover (1) dimensional change due almost entirely to flow, and (2) dimensional change due to a combination of flow and shrinkage caused by loss of water or other volatile matter. The word flow as used in these methods may describe either plastic or elastic deformation or combinations thereof.

NOTE.—Methyl methacrylate and polystyrene are examples of materials that deform almost entirely by flow. Cellulose acetate, cellulose acetate butyrate, phenolic laminated fiber, and vulcanized fibre are examples of materials that deform by a combination of flow and shrinkage.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by the Administrative Committee on Standards, December 12, 1945.

Prior to their present publication as tentative, these methods were published as tentative from 1941 to 1943. They were adopted as standard in 1943, but were revised and republished as tentative in 1944.

### *Method A. Rigid Plastics*

#### Nature of Test

2. The principle of method A is essentially that of the parallel plate plastometer, namely, a constant force system whereby a test specimen is conditioned, if necessary, and is then placed between the parallel plates of a constant force device and the thickness is observed over a required period at the stipulated temperature or temperatures.

#### Apparatus

3. The apparatus shall consist of the following:

(a) *Testing Machine.*—A machine capable of exerting a constant force of 250 lb., 500 lb., and 1000 lb.  $\pm$  1 per cent between the parallel anvils of the machine, which shall be arranged so that they can be brought into contact with the test specimen before the load is applied. A machine suitable for this test is shown in Fig. 1. A recommended method for calibrating such a device is given in the Appendix. One of the anvils of the machine shall preferably be self-aligning and shall, in order that the load may be applied evenly over the face of the speci-

men, be arranged so that the specimen is accurately centered and the resultant of the load is through its center. The machine shall also be equipped with a dial gage or the equivalent capable of measuring the relative movement of the faces to 0.001 in. or less. A thermometer of the total immersion type shall be suspended in such a manner that the bulb is approximately level with the specimen and not more than 3 in. therefrom.

ing shall be tested in the direction parallel to ram travel.

(b) The specimens for materials made in large sheets, such as phenolic laminated fiber, where moisture absorption characteristics may vary over the entire surface, shall be prepared, unless otherwise specified, in accordance with the sampling method described in Paragraph (c) which averages the effect over the sheet by selecting squares comprising the spec-

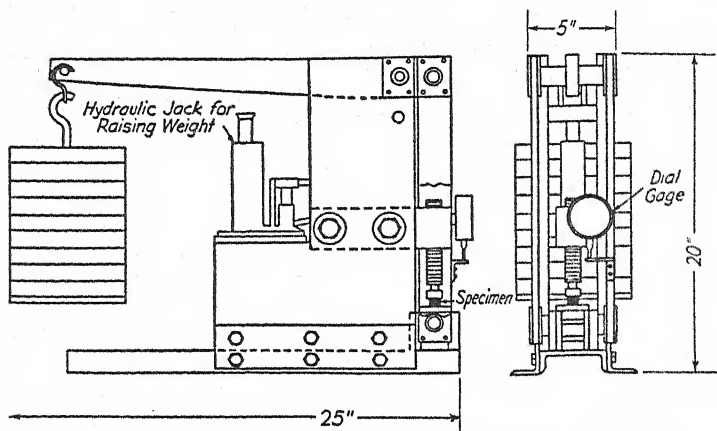


FIG. 1.—Deformation Testing Machine.

(b) *Test Chamber.*—A test chamber of suitable size and construction to enclose the testing machine and maintain it during the test within plus or minus 1 C. (2 F.) of the specified test temperature, except for the short period at the beginning when opening the door may cause a drop in temperature.

#### Test Specimens

4. (a) The test specimen shall be a  $\frac{1}{2}$ -in. cube, either solid or composite. Materials over  $\frac{1}{2}$  in. in thickness shall be reduced to  $\frac{1}{2}$  in., and thinner materials shall be piled up with the total height as near to  $\frac{1}{2}$  in. as possible. The squares of the composite specimen shall be accurately aligned in all cases. Unless otherwise specified, specimens made by mold-

ing shall be tested in the direction parallel to ram travel.

(c) *Specimen from Sheet Materials.*—The specimen shall be selected from the whole sheet by cutting a strip  $\frac{1}{2}$  in. in width from the sheet, parallel to the two long edges if the sheet is not square or parallel to any two if it is square, midway between them, and extending from the edge to the center of the sheet. If quarter sheets are used, the strip shall be taken from the edge corresponding to the center-to-edge section of the original whole sheet. The strip shall be divided into eight equal parts numbered from 1 to 8, beginning with the piece corresponding to the edge of the sheet. The squares used to form the composite

test specimen shall be taken from these pieces. If the whole piece is not used, the squares cut from it shall be taken from the end which was originally nearest the edge of the sheet. If any piece is insufficient for the number of squares required to be cut from a piece bearing that number, a second strip shall be cut adjacent to the first one, cut into pieces, and the required number of squares taken from the appropriate pieces. The number of squares to be cut from each piece is prescribed in Table I.

TABLE I.—NUMBER OF SQUARES TO BE CUT FROM EACH PIECE.

Thick- ness of Mate- rial, in.	Total Number of Squares Required	Number of Piece							
		1	2	3	4	5	6	7	8
$\frac{1}{64}$	32....	8	6	6	4	3	3	2	...
$\frac{1}{32}$	16....	4	3	3	2	2	1	1	...
$\frac{3}{64}$	11....	2	2	2	2	1	1	1	...
$\frac{1}{16}$	8....	2	1	1	1	1	1	1	...
$\frac{3}{32}$	5....	1	1	1	1	...	1	...	...
$\frac{1}{8}$	4....	1	1	1	...	1	...	...	...
$\frac{3}{16}$	3....	1	...	1	...	1	...	...	...
$\frac{1}{4}$	1....	...	...	1	...	...	...	...	...
$\frac{3}{8}$	1....	...	...	1	...	...	...	...	...
$\frac{1}{2}$	1....	...	...	1	...	...	...	...	...

NOTE.—In case of material of thicknesses not included in the above table, the squares used in the composite test specimen shall be selected in accordance with the method for nearest thickness given in the table. In case the thickness is midway between two adjacent values in the table, the squares used in the composite test specimen shall be selected in accordance with the instructions for the thinner material.

### Conditioning

5. (a) Where simple deformation under load is required, without regard to the distinctive effects of flow or shrinkage, or both, and neglecting the influence of elevated service temperatures, the specimens shall be exposed to the standard laboratory atmosphere as prescribed in the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618),<sup>3</sup> and shall be tested under the same conditions, unless otherwise specified.

(b) In those materials where shrinkage

is a large part of total deformation, the specimens shall be preconditioned for 4 hr. at  $65 \pm 3$  C. ( $150 \pm 5$  F.) and then conditioned for 68 hr. at a temperature of  $35 \pm 1$  C. ( $95 \pm 2$  F.) and a relative humidity of  $90 \pm 2$  per cent, unless otherwise specified. The specimens shall be supported during conditioning upon a  $\frac{1}{8}$ -in. mesh wire screen or the equivalent in order to permit free access of the atmosphere to all surfaces.

(c) Where a quick measurement of total deformation is desired, such as for procurement of materials wherein the flow is relatively great compared to shrinkage, the specimens shall be exposed for 16 to 18 hr. in a circulation air oven at 50 C. (122 F.). The specimens shall be removed from the oven and cooled to the standard room temperature (Methods D 618) in a desiccator over anhydrous calcium chloride for a period of at least 3 hr.

(d) Where it is definitely known that the material is not moisture-responsive, conditioning of any kind may be omitted.

### Test Temperatures

6. (a) The test temperature, that is, the temperature of the chamber containing the testing apparatus shall be one or more of the following: 25 C. (77 F.), 50 C. (122 F.), and 70 C. (158 F.), each temperature being maintained within plus or minus 1 C. (2 F.).

### Procedure

7. (a) The conditioned test specimens shall be placed between the anvils of the testing machine immediately upon removal from the conditioning atmosphere. The specimens tested without conditioning shall be at room temperature when placed in the testing machine. Where composite specimens are used, care shall be taken to insure that the squares are well aligned.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) The load shall be applied to the specimen without shock and the initial reading taken as soon as the full load is upon the specimen, as indicated by a sudden slowing of the rate of travel of the gage. At the end of 24 hr., a second reading shall be taken and the total change in height in mils shall be recorded. The original height in mils of the specimen shall be determined by measuring the specimen after it is removed from the testing machine and adding to this the total change in height as read on the dial of the testing machine.

(c) The deformation shall be taken as the percentage change in height of the test specimens after 24 hr., calculated as follows:

$$\text{Deformation, per cent} = \frac{A}{B} \times 100$$

where:

A = change in height in mils in 24 hr.,  
and

B = original height in mils.

#### Report

8. The report shall include the following:

- (1) Original height of test specimen in mils,
- (2) Thickness of components in mils where a composite test specimen is used,
- (3) Conditioning procedure,
- (4) Temperature of test and force applied,
- (5) Change in height of the test specimen in 24 hr. in mils, and
- (6) Deformation (flow or combined flow and shrinkage) expressed as the percentage change in height of the test specimen calculated on the basis of its original height.

#### *Method B. Nonrigid Plastics*

#### Scope

9. This method covers the procedure

for determining the deformation under compression of nonrigid plastic materials approximately 0.5 in. in thickness. The word deformation is used herein in the broad sense to cover the total dimensional change due to compression, both elastic and plastic.

#### Nature of Test

10. Method B is essentially the same as method A except that the pressure is 100 psi., and the period of test used for deformation is 3 hr. The recovery is based on removing the specimen from compression and allowing it to remain at the stated temperatures for a period of 1 hr. and at room temperature for  $\frac{1}{2}$  hr., after which the amount of recovery is measured.

#### Apparatus

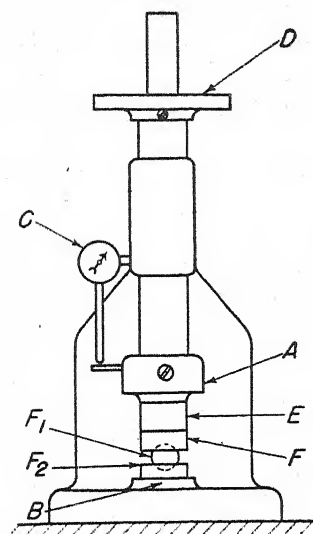
11. The apparatus shall consist of the following:

(a) *Testing Machine*.—A testing machine similar in principle and operation to the one described in method A (Section 3 (a)), except that it shall be capable of exerting a constant force of 100 lb.  $\pm$  1 per cent. A machine suitable for this test is illustrated in Fig. 2. The machine shown in Fig. 1 may be used provided it is equipped with a locking device for the upper anvil so as to obtain a zero point. A lock nut added to the upper portion of the movable anvil has been found suitable for this purpose.

NOTE.—In making tests in accordance with method B, care should be taken to reduce machine friction to a minimum. This is particularly desirable when using the high-pressure machine shown in Fig. 1.

(b) *Test Chamber*.—A test chamber similar to that prescribed in method A (Section 3 (b)).

(c) *Conditioning Chamber*.—A chamber for drying, consisting of a desiccator provided with anhydrous calcium chloride.



A—Movable anvil.  
B—Stationary plate.  
C—Thickness indicator.  
D—Weight platform.  
E—Test specimen.  
F—Lower anvil mounted on ball support (F<sub>1</sub>) and loose plate (F<sub>2</sub>).

FIG. 2.—Low-Pressure Deformation Tester.

### Test Specimens

12. The test specimens shall be 1.129 in. in diameter by  $\frac{1}{2}$  in. in thickness, this size being suitable for the high-pressure testing machine shown in Fig. 1, whose arm without weights but including pan provides a load of 100 lb. (Note 1). A single specimen shall be used. The specimen shall be molded or cut (Note 2) from a  $\frac{1}{2}$ -in. sheet at least 1 in. from the edges and so selected as to be of uniform thickness, homogeneous, and free from bubbles.

NOTE 1.—Unavoidable variations in diameter of the specimen may require slight adjustment of load to obtain 100 psi.

NOTE 2.—A tool suitable for this purpose is described in the Standard Methods of Test for Compression-Deflection Characteristics of Vulcanized Rubber (A.S.T.M. Designation: D 575).<sup>3</sup>

### Conditioning

13. (a) The specimen shall be con-

ditioned by placing it in the desiccator for 24 hr., unless it is definitely established that the material is not affected by moisture. The specimen shall be so supported and desiccated as to permit free access of the atmosphere to all faces and edges.

(b) The test temperatures, that is, the temperatures of the chamber containing the testing apparatus, shall be one or more of the following: 25 C. (77 F.), 50 C. (122 F.), and 70 C. (158 F.), each temperature being maintained within plus or minus 1 C. (2 F.).

### Procedure

14. The procedure for method B shall be the same as that of method A, as described in Section 7, except that the duration of the deformation test shall be 3 hr. Also the zero point or initial reading for the beginning of the test shall be obtained by determining the dial reading with the anvils together under full load and calculating the zero point from the thickness of the specimen measured with a micrometer having a low-pressure ratchet attachment and capable of measuring to the nearest 0.2 mil. After determining the thickness of the specimen at the end of 3 hr. the specimen shall be removed from the machine and left for 1 hr. in the chamber at the temperature at which it is being tested. It shall then be removed from the chamber and kept at room temperature for  $\frac{1}{2}$  hr. after which the thickness shall be determined again with a micrometer.

### Calculations

15. The deformation and recovery shall be calculated as follows:

$$\text{Deformation, per cent} = \frac{H_0 - H_1}{H_0} \times 100$$

$$\text{Recovery, per cent} = \frac{H_2 - H_1}{H_0 - H_1} \times 100$$



where:

$H_0$  = original specimen height or thickness,

$H_1$  = specimen thickness after 3 hr. under load of 100 psi., and

$H_2$  = specimen thickness  $1\frac{1}{2}$  hr. after load removal.

### Report

16. The report shall include the following:

(1) Temperature of test,

(2) Original thickness of test specimen in mils,

(3) Total deformation in 3 hr. expressed as a percentage change in thickness calculated on the basis of its original thickness, and

(4) Recovery, the total increase in specimen height which occurs during the  $1\frac{1}{2}$ -hr. period following load removal, expressed as a percentage of the original deformation.

## APPENDIX

### CALIBRATION OF DEFORMATION-UNDER-LOAD TESTING MACHINE PER METHOD A

The deformation-under-load testing machine may be calibrated by means of a calibrating bar and a proving ring, as shown in Fig. 4. Figure 3 shows a calibrating bar with dimensions suitable for use in the testing machine illustrated in Fig. 1.

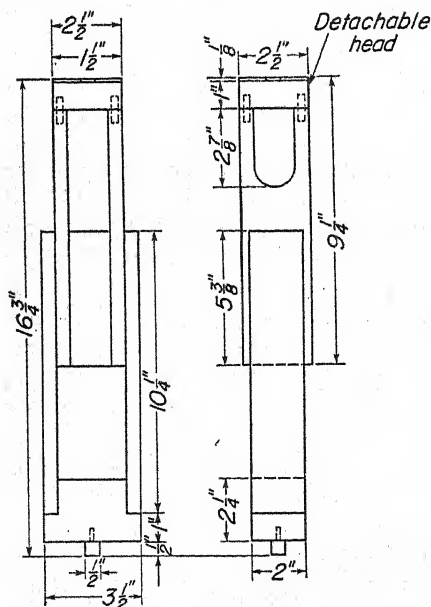


FIG. 3.—Bar for Calibrating Deformation-Under-Load Testing Machine.

The dial gage, its actuating detail, the upper anvil and screw and the jack control handles should be removed to make room for the calibrating bar which, when properly in place, will be in contact with only the lower anvil of the load deformation machine. The weight of the

calibrating bar and the proving ring should be determined and added to the load indicated by the proving ring. The testing machine with the calibrating bar and the proving ring should be set up in a compression testing machine or other suitable machine which has sufficient opening and is controllable to move the upper contact of the proving ring up or down or hold it stationary, as required. To calibrate the machine

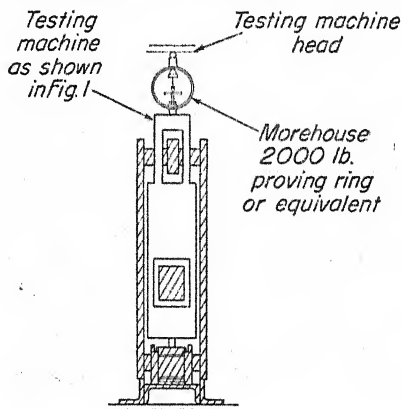


FIG. 4.—Arrangement of Bar and Proving Ring in Calibration of Testing Machine.

for any load the weights to be used for giving that load should be placed on the machine. Readings of the proving ring may be made with the compression machine held stationary or with the proving ring and test bar moving either up or down. Movement at a rate of a few mils per second is easy to attain and provides sufficient time for measurement. For a complete calibration it is desirable to measure the loads with the machine both moving and stationary in the upper, middle, and lower positions.

# *Tentative Method of Test for*

## DEVIATION OF LINE OF SIGHT THROUGH TRANSPARENT PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 881-46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method describes a procedure for measuring, by means of a telescope and a target, the deviation of the line of sight through flat or curved sections made of transparent plastics.

### Significance of Test

2. This is a functional test for sheets or formed articles, such as certain aircraft windshields and enclosures, the manner of use of which requires information about the deviation of the line of sight. The property measured is determined jointly by the prismatic power of the plastic at each point and by the angle made with the surface at that point by the line of sight. Thus the measurement should be considered as a specialized procedure capable of excellent precision and reproducibility but not applicable for determining optical distortions nor for defining optical quality in any general sense.

### Outline of Method

3. A line of sight is established by focusing a telescope on a target. By placing the specimen to be tested in the line of sight, the apparent position of the cross hairs on the target is shifted. From the magnitude of the shift and the distance between the target and the specimen, the angular deviation of the line of sight by the specimen is calculated.

### Apparatus

4. (a) *Telescope*.—A rigidly mounted telescope equipped with cross hairs shall be provided (Note 1). The telescope shall be sufficiently free of parallax so that when the eye of the observer is shifted, the corresponding movement of the image of the target relative to the cross hairs shall not exceed one tenth of the smallest scale division of the target. The magnifying power of the telescope shall be adequate to permit accurate reading of the position of the cross hairs on the target. The mounting of the tele-

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

scope shall be such that it cannot be displaced readily after being clamped in position. To obtain clear images through some specimens, it may be necessary to stop down the objective of the telescope (Note 2).

NOTE 1.—An engineer's transit is a very suitable instrument for measuring deviations of line of sight.

NOTE 2.—The intended use of the article or material under test may determine the size of stop to be used. Thus, if the user will look through the material with his unaided eye, then the diameter of the stop should approximate that of the pupil of the eye. Larger apertures make for increase of brightness of the image, but frequently the definition is poorer and the measurements are more difficult or more fatiguing.

(b) *Target*.—A target with a suitable scale shall be provided. The markings shall be so made and the illumination shall be such that the divisions shall be clearly visible when viewed through the telescope (Note 3). Where deviation in one direction only is of interest, the target may consist of parallel straight lines; otherwise the most convenient form consists of concentric circles.

NOTE 3.—Lines ruled in black on a white background, which may be either opaque with front lighting or translucent with back lighting, make the most suitable target for most purposes. The spacings of the lines can be calculated by a formula as follows:

$$s = \frac{\theta L}{3440}$$

where:

$s$  = the spacing of the lines, in inches,

$\theta$  = the angular deviation, in minutes, corresponding to an apparent shift of the cross hairs from one cross line to the next line, and

$L$  = the distance of the target from that part of the test specimen which is under examination, in inches.

Thus, if the distance of specimen to target is 40 ft., and if an apparent shift of the cross hairs from one line to the next is to correspond to one minute of angular deviation by the specimen, the spacing of the lines will be:

$$s = \frac{L \times 40 \times 12}{3440} = 0.140 \text{ in.}$$

(c) *Support for Specimen*.—A suitable fixture for support of the specimen shall be provided (Note 4). The specimen shall be firmly held during each measurement, and means shall be provided for moving the specimen quickly to each specified position.

NOTE 4.—Small flat sheets may be supported by hand while measurements are being made. Large flat sheets and curved articles require special fixtures, the design of which is dictated by the specific optical requirements of each article.

### Procedure

5. The telescope shall be adjusted so that the cross hairs coincide with a reference point or line on the target, and shall be firmly locked or clamped in this position. Great care shall be taken not to displace the telescope or target during the measurements, and the initial setting shall be rechecked frequently. The specimen shall be placed in the prescribed position. Unless otherwise specified, the specimen shall be so placed and so oriented that the line of sight shall intersect its surface normally (Note) at a point 12 in. from the objective lens of the telescope. The position of the cross hairs on the image of the target shall be recorded if deviations at specific points are to be taken. If maximum deviations only are sought, the observer shall note continuously the apparent position of the cross hairs on the target while the sheet or article is moved through the line of sight until the entire specified area has been covered, and shall record the largest apparent displacement of the cross hairs from the reference point or line.

NOTE.—The deviation of the line of sight is determined both by the angle of incidence and by any lack of parallelism of the surfaces. If the line of sight intersects the surface normally, any deviation is due to lack of parallelism, such as may be caused by "thinning out" during

shaping operations. In actual use, however, the line of sight is rarely normal to the surface and the usual practice is to test aircraft enclosures and similar articles in such a way that the eye of the observer and his line of sight duplicate the position and direction of those of the ultimate user.

### Calculations

6. The angular deviation of the line of sight, in minutes, produced by the specimen is given by the following formula:

$$a = N\theta$$

where:

$N$  = the number of rulings by which the apparent position of the cross hairs has been shifted, and

$\theta$  = the angular deviation corresponding to the distance between adjacent rulings.

### Report

7. Unless otherwise specified the report shall include the following:

- (1) Nature of article tested,
- (2) Angle of intersection of line of sight with surface, if this is constant, or description of the arrangement used, and
- (3) Deviation of line of sight at specified points, with the direction of the deviation relative to a prescribed direction on the specimen, or maximum deviation of line of sight.

# *Tentative Method of Test for*

## COEFFICIENT OF CUBICAL THERMAL EXPANSION OF PLASTICS<sup>1</sup>



• A.S.T.M. Designation: D 864 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method of test is intended for the determination of the coefficient of cubical thermal expansion of both rigid and nonrigid plastics.

(b) The thermal expansion of a plastic is composed of a reversible component on which are superimposed changes in volume due to changes in moisture content, curing (degree of polymerization), loss of plasticizer or solvents, release of stresses, and other factors. This method of test is intended for determining the reversible cubical thermal expansion under exclusion of these spurious factors. Some plastics show more or less abrupt changes in the coefficient of cubical thermal expansion in narrow temperature intervals, which indicate changes in molecular structure or second order transition points. They can be investigated by careful measurements in the critical temperature range.

### Apparatus

2. The apparatus shall consist of the following:

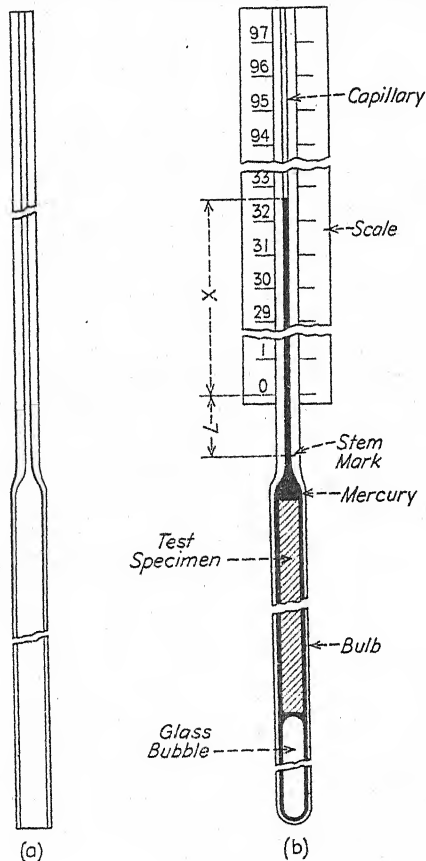
(a) *Dilatometer*.—A glass dilatometer as shown in Fig. 1 (Note). It shall consist of two pieces of Pyrex glass tubing sealed together as shown in Fig. 1 (a). The lower or bulb member shall be approximately 20 cm. in length, 10 mm. in outside diameter, and 1 mm. in wall thickness. The capillary part shall be approximately 1 m. in length, 6 mm. in outside diameter, and about 0.7 mm. in inside diameter. The inside diameter shall be tested for uniformity and, if necessary corrections shall be made for irregularities in diameter.<sup>3</sup> A suitable scale shall be attached to the dilatometer as shown in Fig. 1 (b). The scale shall extend from the top of the capillary to within a short distance of the stem mark and shall be graduated so that readings can be made to the nearest 0.05 cm.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Administrative Committee on Standards, December 12, 1945.

<sup>3</sup> For method of calibrating capillary see Joseph Reilly and W. N. Rae, "Physico-Chemical Methods," Vol. I, pp. 362 to 365, Van Nostrand, New York (1943).

NOTE.—Glass dilatometers as described in this method have been used successfully. Dilatometer bulbs made from a metal with low thermal expansion may also be used, provided the expansion characteristics of the metal are known and are taken fully into consideration when calculating the coefficient of cubical thermal expansion of the specimen. It is hoped that a satisfactory design of a metal dilatometer can be shown in a future revision of this method.



NOTE.—The distance from the stem mark to the neck of the bulb shall be about 2 cm.

FIG. 1.—Dilatometer.

(b) *Temperature Bath*.—A well-agitated liquid bath for controlling the temperature of the specimen.

NOTE.—The following baths have been found useful:

Range	Bath Liquid
-35 to +10 C.....	solid carbon dioxide - acetone, controlled to $\pm 0.2$ C.
+10 to +90 C.....	water controlled to $\pm 0.2$ C.
+90 to +150 C.....	oil controlled to $\pm 0.2$ C.

(c) *Thermometer*.—A thermometer to determine the temperature of the liquid bath with an accuracy of plus or minus 0.1 C.

### Test Specimens

3. (a) The samples from which test specimens are to be cut or shaped shall be prepared by methods and under conditions which insure the absence of voids and gas bubbles, and which yield specimens that are substantially free of stresses. This requirement applies also if the specimens are molded to their finished shape.

(b) The test specimen shall be a rod approximately 15 cm. by 6 by 6 mm. with rounded edges. It may consist of two or more pieces although a single piece is preferred.

NOTE.—Dimensions of specimen, bulb, and capillary may be changed to suit special conditions. In general, results will be more accurate if ratio of mercury volume to specimen volume is small and if ratio of meniscus travel to temperature difference is large.

### Conditioning of Specimens

4. For those tests where conditioning is required, the test specimens shall be conditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>4</sup> With materials whose water absorption is known to be negligible, the conditioning may be omitted.

### Procedure

5. (a) Determine the weight,  $W$ , in grams, of the specimen, and its volume,  $V$ , in cubic centimeters, by direct measurements or by means of specific gravity. Seal the test specimen into the bulb as shown in Fig. 1 (b). Place a Pyrex glass bubble in the bottom of the bulb to prevent excessive heating of the specimen during sealing. Determine the weight,  $W_1$ , of the tube containing the sealed-in specimen.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) Fill the tube with mercury by a vacuum technique, and adjust the level of the mercury meniscus by introducing a clean piano wire to remove excess mercury. This adjustment shall be made so that at the lowest and highest temperatures to be used the meniscus stays within the range of the scale attached to the dilatometer. Determine the weight,  $W_2$ , of the tube containing the specimen and the mercury.

(c) Immerse the apparatus in the temperature bath up to the stem mark, and adjust the temperature of the bath to the lowest point of the range to be investigated, maintaining the temperature at that point until the position of the mercury meniscus,  $X$ , has remained constant for 15 min. Increase the bath temperature in increments of 5 or 10 C., taking corresponding readings of the meniscus position.

(d) An alternative heating schedule which is usually more rapid may be used, and consists of continuously varying the bath temperature at a maximum rate of 0.5 C. per min. (Note). Readings of the position of the mercury meniscus,  $X$ , shall be taken at increments of 5 C.

NOTE.—If the bulb or the specimen are of a larger diameter than prescribed in Section 2(a) or 3(b) it may be necessary to reduce the rate of temperature rise in order to avoid excessive temperature lag between the bath and the specimen.

(e) Make duplicate determinations with rising temperature and in case of disagreement between these two determinations make a third or fourth determination until duplicate values are obtained.

NOTE.—Values obtained on the first determination may disagree from values obtained on subsequent determinations because the specimen may have contained stresses which have been released at the temperatures reached during the first determination.

## Calculation

6. (a) The position of the mercury meniscus shall be plotted against temperature on linear coordinate paper. In general, this curve will consist of two straight line portions which intersect at the transition temperature. The slope of each straight-line portion shall be determined and this shall form the basis for calculating the coefficient of cubical thermal expansion above and below the transition temperatures.

(b) Determine the internal volume  $V_o$  of the bulb below the stem mark as follows:

$$V_o = \frac{W_2 - W_1}{S_m} + V - (X_o + L)A$$

where:

$W_2 - W_1$  = total weight of mercury in grams,

$S_m$  = density of mercury (13.53 g. per cu. cm.),

$V$  = volume of test specimen in cubic centimeters,

$X_o + L$  = distance of mercury meniscus from stem mark in centimeters at time of weighing, and

$A$  = cross-section of capillary bore in square centimeters.

(c) Calculate the coefficient of cubical thermal expansion, both above and below the transition temperature, as follows:

$$a = \frac{A \Delta X}{V \Delta t} - \frac{V_o}{V} (a_m - a_o) + a_m$$

where:

$a$  = coefficient of cubical thermal expansion of specimen,

$A$  = cross-section of capillary bore, in square centimeters,

$V$  = volume of test specimen, in cubic centimeters,

$V_o$  = internal volume of bulb below stem mark, in cubic centimeters,



$a_m$  = coefficient of cubical thermal expansion of mercury ( $18.2 \times 10^{-5}$ ),

$a_g$  = coefficient of cubical thermal expansion of Pyrex glass ( $1.0 \times 10^{-5}$ ), and

$\frac{\Delta X}{\Delta t}$  = slope of a straight line portion of the plot determined from two points  $(X_1, t_1)$  and  $(X_2, t_2)$  where  $\Delta X = X_2 - X_1$  and  $\Delta t = t_2 - t_1$ .

## Report

7. The report shall include the following:

(1) Designation of material, including name of manufacturer and information on composition when known,

(2) Method of preparation of test specimen,

(3) Temperature range over which the measurements were made,

(4) Coefficient of cubical thermal expansion per degree Centigrade.

(5) If a transition point is found, include the transition temperature and the coefficients of cubical thermal expansion below and above the transition temperature,

NOTE.—For data to be published in tables of properties of plastics, it is recommended that the coefficient of cubical thermal expansion as derived from readings at  $-30$  C. and  $+30$  C. be given.

(6) Complete description of any unusual behavior of the specimen.

# *Tentative Method of Test for*

## FLAMMABILITY OF PLASTICS, SELF-EXTINGUISHING TYPE<sup>1</sup>



A.S.T.M. Designation: D 757 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is designed to evaluate the flammability of rigid plastics in the form of sheets or plates  $\frac{1}{8}$  in. in thickness when brought into contact with a surface at bright red heat. It is designed to be used for materials found to be self-extinguishing when tested by the Standard Method of Test for Flammability of Plastics Over 0.050 in. in Thickness (A.S.T.M. Designation: D 635).<sup>3</sup>

NOTE.—For tests of plastics in the form of thin sheets or films 0.050 in. and under in thickness, reference should be made to the Standard Method of Test for Flammability of Plastics 0.050 in. and Under in Thickness (A.S.T.M. Designation: D 568).<sup>3</sup> Flammability tests of sheet and plate insulation are covered in the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229).<sup>3</sup>

### Apparatus

2. The apparatus shall consist of the following:

(a) *Test Chamber*.—A room or enclosure protected from air currents, but provided with means for venting the fumes from burning specimens. A hood may be used if its exhaust fan is turned off during the test and allowed to run only periodically to clear out the fumes between tests.

(b) *Test Unit*.—A test unit consisting of the parts shown in Figs. 1 and 2. The igniting source, *A*, shall be a globar element<sup>4</sup> supported in upright holders, *B*, with ceramic or asbestos bushings. The globar element shall be heated to 950 C. (1742 F.) by alternating or direct current. By means of a voltage regulator, or rheostat, and suitable voltmeter and ammeter or a wattmeter, the electrical input shall be adjusted to  $350 \pm 20$  w. On typical globar units of this type, this

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by Committee E-10 on Standards, January 19, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> Such a globar element is obtainable from the Globar Division, The Carborundum Co., Niagara Falls, N. Y., and is designated as their type "AT," 11 by 4 by 5/16 in. (The overall length is 11 in., the length of the high resistance portion is 4 in., the diameter is 5/16 in.) The assembly of the element requires two No. 2100 type M clamps and two No. 2051 terminal straps.

variation of power input will give a range of temperature from 900 to 1000 C. (1652 to 1832 F.) which will not signifi-

### Procedure

4. (a) The igniting bar shall be heated to the test temperature of 950 C.

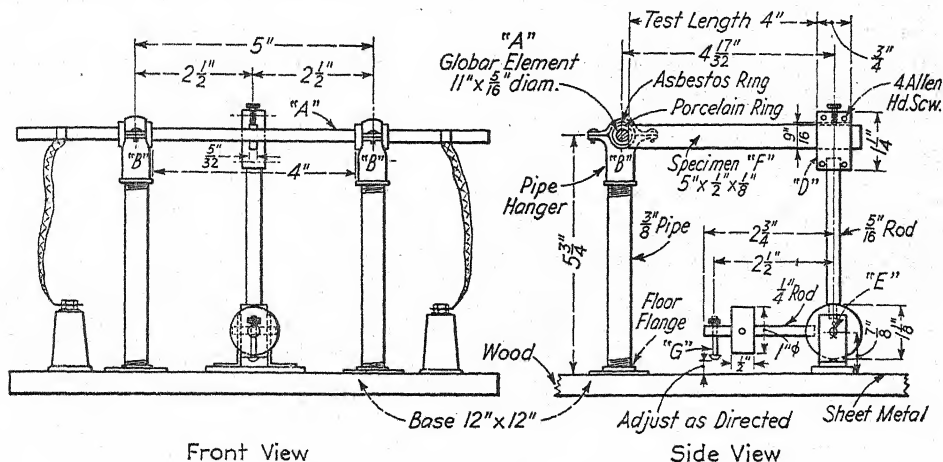


FIG. 1.—Flammability Test Apparatus.

cantly affect the results of the test. If available, an optical pyrometer may be used to check the temperature of the bar. When at the proper temperature, the bar should glow a bright cherry red. The specimen holder, *D*, shall be pivoted on the base at *E*, to permit the specimen, *F*, to remain in contact with the igniting bar to the extent of 0.200 in. by spacing of the stop screw, *G*. A 20-mesh gauze screen for catching any molten material shall be located 0.25 in. below the lowest position of the lower edge of the specimen and shall be supported as shown in Fig. 2.

(c) *Stop Watch or Clock*.

### Test Specimens

3. At least three test specimens 5 in. in length by  $\frac{1}{2}$  in. in width by  $\frac{1}{8}$  in. in thickness shall be tested. They may be sawed, cut, or milled from sheet stock or from standard test bars. The thickness shall not vary more than 0.010 in. from the nominal  $\frac{1}{8}$  in. Specimens shall be tested in the as-received condition. Special preconditioning of the specimens may be agreed upon by the purchaser and the manufacturer.

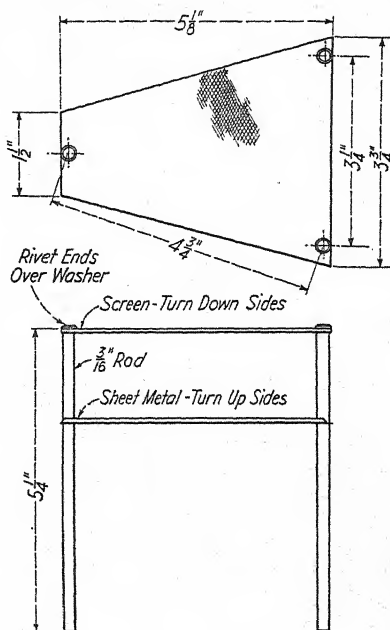


FIG. 2.—Screen Ash Tray.

(1742 F.) as described in Section 2 (b) and allowed to heat until the electrical meters indicate a steady current.

(b) The specimen shall be clamped in

the specimen holder with its length horizontal and at right angles to the axis of the igniting bar and its width in a vertical plane. The length free to burn outside of the clamp shall be 4 in. The front edge of the clamped specimen shall be brought into contact with the igniting bar and allowed to remain in this position for 3 min.

NOTE.—Easily flammable materials will burn or melt their way to the stop screw, *G*, within a few seconds. The less flammable materials will burn or char only a small area adjacent to the igniting bar.

(c) After 3 min. any flaming shall be extinguished, the specimen removed, and the length burned shall be measured. For convenience, the burned specimen may be placed beside an untested speci-

men and the length of the burned specimen measured to the nearest 0.05 in. at the center of the  $\frac{1}{2}$  in. side, to the point at which no charring or melting is visible. This length divided by three shall be recorded as the rate of burning in inches per minute. If a material burns up to the clamp in less than 3 min., the time of burning in minutes shall be noted, and divided into four to give the rate of burning in inches per minute.

### Report

5. The burning rate in inches per minute shall be reported as the average of at least three tests. In case the specimen melts without burning, or shows any unusual behavior, this should be recorded.

# Tentative Method of Test for REPEATED FLEXURAL STRESS (FATIGUE) OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 671 - 42 T

ISSUED, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. This method covers the test procedure for determining the effect of repetitions of stress on plastics by a fixed-cantilever type of testing machine designed to produce the same maximum deflection of the specimen in each cycle (Note). This test is also applicable to plastic impregnated wood and plastic bonded plywood.

NOTE.—Since the characteristics which render a material resistant to fatigue are not completely known, and since the effect of such factors as the variations in shape of specimen, type of testing machine, and conditions of test (speed of machine, temperature of specimen, atmospheric temperature, and relative humidity), are not completely known, the details considered in this method concern those factors known to have important influences. Since other factors equally as important may in the future be discovered, the details of this tentative method are largely advisory in nature.

## Definitions

2. (a) *Fatigue (Progressive Fracture)*.—The phenomenon of the progressive fracture of a material by means of a

crack which begins and spreads under the action of repeated cycles of stress.

(b) *Fatigue Strength ( $S_F$ )*.—The maximum amplitude of an alternating stress cycle, expressed in pounds per square inch, which will not cause fracture of the material for a given number of cycles of alternating stress. The corresponding number of cycles of stress must be stated (Note). If the term is used without further qualification, it is understood that the stress cycle is such as to produce a complete reversal of stress (from tension to compression), see Fig. 1 (a). When the stress cycle does not produce complete reversal of stress the mean stress of the cycle must also be stated. (In this case the fatigue strength is not the maximum stress in the cycle, Fig. 1 (b), (c), (d)).

NOTE.—For some plastics and some metals the fatigue strength remains constant beyond a certain number of cycles of stress. That is, below a certain value of alternating stress the material does not fracture for an indefinitely large number of cycles of stress. The corresponding value of fatigue strength is commonly known as the endurance limit.

Unfortunately, in the literature on fatigue of metals the terms endurance limit, endurance

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1942.

strength, and fatigue limit have been used interchangeably. They have been used to refer to the *maximum stress* in a stress cycle below which no evidence of fracture can be detected after a very large number of cycles of loading. The same terms have also been used to refer to the maximum stress in a stress cycle which would cause fracture after an arbitrarily selected number of cycles. This definition has been used in tests of metals for applications in which the number of repetitions of maximum loading in the life of the structure was expected to be relatively small.

(c) *Mean Stress ( $S_M$ )*.—The algebraic mean between the maximum and mini-

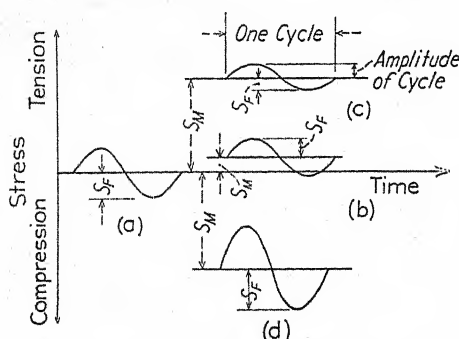


FIG. 1.—Stress-Time Curves for Different Ranges of Variations of Stress.

mum stress produced in a material during an alternating cycle of stress. When used in conjunction with the fatigue strength the term, mean stress, denotes the mean stress for which the stated fatigue strength was determined. The sign of the mean stress, whether tension (+) or compression (−), must be stated. The mean stress is used together with the fatigue strength to define the range of variation of stress in the stress cycle for which the fatigue strength was determined. The mean stress of a cycle is important because in general the fatigue strength of a given material will depend upon the magnitude of the mean stress. Thus it is necessary to specify both the fatigue strength and the mean stress.

In Fig. 1 stress *versus* time curves are shown for one cycle of stress with differ-

ent combinations of mean stress  $S_M$  and superimposed alternating stress. The alternating stress cycle shown has an amplitude equal to the fatigue strength  $S_F$ .

In Fig. 1 (a) the fatigue strength is  $S_F$ , the mean stress is zero, and the maximum tension equals the maximum compression.

In Fig. 1, (b), (c), (d) the mean stress  $S_M$  is not zero. In Fig. 1 (b) the cycle of stress produces a maximum tension greater than the maximum compression. In Fig. 1 (c) all values of stress in the cycle are tension. In Fig. 1 (d) all values of stress in the cycle are compression.

NOTE.—Another way of defining the range of variation of stress in a stress cycle is to use the “range ratio.” The range ratio has been defined as the ratio of the (numerical) minimum stress to the maximum for any cycle. Thus the range ratio and the fatigue strength must be stated to define the range of variation of stress in the stress cycle for which the fatigue strength was determined.<sup>3</sup>

(d) *Fatigue Ratio*.—The ratio of fatigue strength for cycles of completely reversed stress to the static tensile strength of the material as determined in accordance with the Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638) of the American Society for Testing Materials.<sup>4</sup>

## Apparatus

3. The apparatus (Fig. 2) shall consist of the following:

(a) *Testing Machine*.—A fatigue testing machine of the fixed-cantilever, repeated constant-deflection type (see Appendix I). In this machine the specimen, A, Fig. 2, shall be held as a cantilever beam in a vise, B, at one end,

<sup>3</sup> See “Present-Day Experimental Knowledge and Theories of Fatigue Phenomena in Metals,” Appendix to the Report of Research Committee on Fatigue of Metals, *Proceedings*, Am. Soc. Testing Mats., Vol. 30, Part I, p. 260 (1930).

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

$M$ , and bent by a concentrated load applied to a holder,  $J$ , fastened to the other end. The bending shall be accomplished by a connecting rod,  $C$ , driven by a variable eccentric,  $D$ , mounted on a shaft. The shaft shall be rotated at constant speed by a motor. The vise may be set in the plane of the eccentric so that the beam will be deflected the same amount on either side of the neutral position (completely reversed stress) or the vise may be set so that the

deflection is greater on one side than on the other (mean stress not zero).

(b) *Counter*.—A counter,  $K$ , Fig. 2, geared to the shaft so as to record the number of cycles. A suitable mechanically or electrically operated cut-off switch,  $L$ , shall be provided to stop the machine when the specimen fractures.

(c) *Holder*.—A holder,  $J$ , Fig. 2, for the test specimen proportioned in such a way as to place the center of percussion of the oscillating portion of the specimen

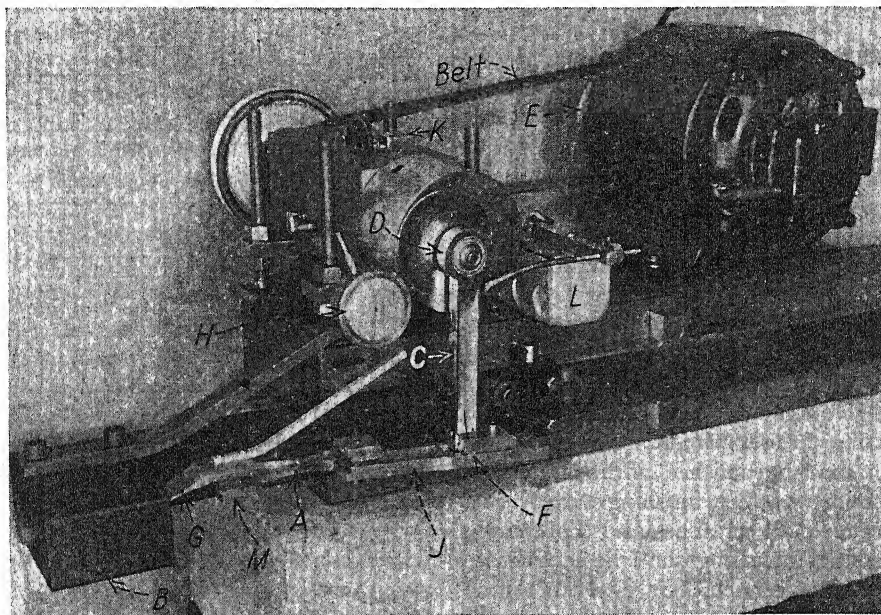


FIG. 2.—Fixed-Cantilever, Repeated-Constant-Deflection Type Fatigue Testing Machine.

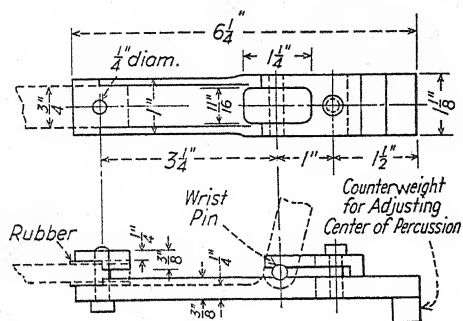


FIG. 3.—Holder for Test Specimen.

and holder at the wrist pin,  $F$ . It shall have as little mass and as large rigidity as possible in order to minimize inertia effects. A diagram of the holder for the free end of the specimen is shown in Fig. 3. It is necessary, for some materials, to use rubber gaskets in the grips on both the holder and the dynamometer to prevent failure at the grip due to stress concentration and chafing.

(d) *Dynamometer*.—The vise,  $B$ , Fig. 2, containing a dynamometer (Note



for measuring the load on the specimen. The dynamometer shall consist of a reduced section, *G*, which acts as a spring, and a dial gage, *H*, to measure the deflection of the spring when the specimen is deflected.

NOTE.—Another form of this machine does not contain a separate dynamometer but uses the specimen as a dynamometer. In this case a dial is used to measure the deflection of the specimen under a known load, preferably the load which is repeatedly applied to the specimen. This requires a separate calibration for each specimen. The result obtained is very nearly identical to that obtained with a dynamometer.

### Test Specimens

4. (a) *Unnotched Specimen*.—The unnotched specimen shall conform to the dimensions shown in Fig. 4 (a). The cross-section at the center of the specimen shall be 0.3 in. square, except in the case of the specimen from thin sheet<sup>5</sup> which shall be 0.3 in. by the thickness of the sheet. The 2-in. radius (Note) of the scalloped sides shall be formed by milling, using a very sharp cutter and such combination of speed and feed as will give a good finish with a minimum of heating of the specimen. The test specimen shall be polished with successively finer emery paper, finishing with No. 00 to remove all scratches and tool marks. The final polishing shall be lengthwise of the specimen, since even small scratches transverse to the direction of tensile stress tend to lower the fatigue strength. In order to avoid heating, all polishing shall be done either by hand or with light pressure on a slowly revolving sanding drum. Care shall be taken to avoid rounding the corners.

<sup>5</sup> For the purposes of this test a thin sheet shall be defined as a sheet less than 0.3 in. in thickness or a material for which the ratio of the modulus of elasticity to the endurance limit is less than 100. The reason for these restrictions is that thin sheets and materials having a low modulus of elasticity are bent so much under the required loads that the fatigue specimen cannot (in the deflected position) be considered a straight beam and hence the equation  $s = \frac{Mc}{I}$  is not accurate. It should be noted that the grips (Fig. 3) may not be satisfactory for specimens of very thin sheet due to the inertia forces set up by the vibration.

NOTE.—With a cantilever specimen the maximum stress does not occur exactly at the minimum section, but slightly farther from the point of loading, *F*, Fig. 2. However, with a moment arm of 5 in. and a specimen with a radius of 2 in. this discrepancy is small enough to be neglected.

(b) *V-Notched Specimen*.—The V-notched specimen for testing materials other than thin sheets shall conform to the dimensions shown in Fig. 4 (b). The standard V-notch shall be at the center of the specimen on one side and shall be 0.01 in. in radius and 0.02 in. in

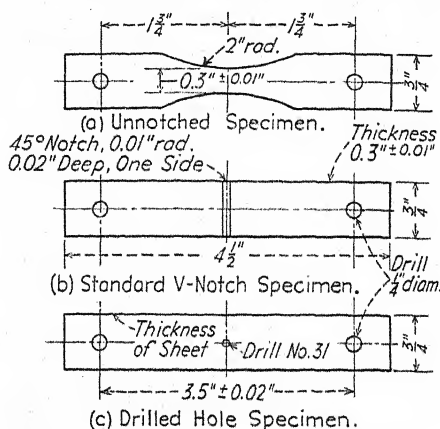


FIG. 4.—Specimens for Fatigue Test.

NOTE.—In the case of unnotched specimens the thickness of the specimen shall be  $0.3 \pm 0.01$  in., except unnotched specimens for sheets which shall be the thickness of the sheet.

depth. The notch shall be formed by milling or shaping in such a way that it will be free from scratches. After machining, all edges of the specimen shall be smoothed to remove burrs.

(c) *Drilled-Hole Notched Specimen for Thin Sheets*.—The notched specimen for testing thin sheets<sup>5</sup> shall conform to the dimensions shown in Fig. 4 (c). The notch shall consist of a transverse hole drilled with a sharp No. 31 drill, 0.120 in. in diameter. The specimen shall be polished in the region of the hole before the hole is drilled and all burrs shall be removed after drilling. No attempt

shall be made to polish the inside of the hole.

#### Possibilities and Limitations of Test

5. (a) The fatigue test is inherently a time-consuming test and not convenient for acceptance testing. Its primary function is in the evaluation of fatigue strength of materials and determination of data for design.

(b) The type of machine covered in this recommended practice is suitable for determining the fatigue strength of both notched and unnotched specimens for any value of mean stress in *tension*.

(c) Specimens notched on one side (as in Fig. 4(b)) may be used to determine the notched fatigue strength for any value of mean stress in either tension or compression. It should be mentioned that for material (such as cellulose acetate) which creeps rapidly, the effect of a mean stress other than zero is to cause relaxation so that the stress cycle tends to approach the condition of complete reversal of stress.

(d) The notched specimen is used to determine the effect of a notch of standard dimension upon the fatigue strength of the material. The V-notch considered in this test as standard is shown in Fig. 4(b). The transverse-hole notch shown in Fig. 4(c) may be used for thin sheet and material such as plywood for which the continuity of the material would be altered by the V-notch.

(e) In any plastic part there are likely to be changes in cross-section, such as fillets, holes, screw threads and the like, at which the state of stress is complex (a combination of normal or shearing stresses, or both, in more than one direction) and at which the maximum value of stress is greater than that calculated by formulas such as  $S = \frac{Mc}{I}$ .

It is known that the effect of a certain type of notch in reducing the fatigue

strength of one material may be more severe than in another material; the use of a standard V-notch in a fatigue test specimen is therefore desirable as a means of evaluating the sensitivity of a material to the effect of a stress raiser.

(f) Other notches have been proposed to replace those shown in Fig. 4(b) and (c) but experimental evidence of their suitability is at present lacking.

(g) Tests of thin sheet<sup>5</sup> yield results which vary with the thickness of the sheet even though the material is identical.<sup>5</sup> Because of this fact the thickness of the sheet shall be specified when reporting results of tests of thin sheet; and all comparisons of different materials, or selection of materials on the basis of fatigue strength shall be made from results of tests of standard specimens or tests in which the same thickness of sheet is used for all materials.

(h) In the application of this test procedure it is assumed that test specimens of a given material are comparable and truly representative of the material. Departure from this assumption may introduce discrepancies as great as, if not greater than, those due to departure from details of procedure outlined in the test.

#### Conditioning

6. (a) *Preconditioning*.—All specimens shall be conditioned for 14 days in the atmosphere of the enclosure in which the tests are to be conducted.

(b) *Test Conditions*.—Tests shall be made in a room maintained at a constant temperature of  $77 \pm 2$  F. ( $25 \pm 1.1$  C.) and relative humidity of  $50 \pm 2$  per cent except when it is desired to evaluate the effect of high or low temperature or relative humidity on the endurance limit. Under all circumstances the temperature and relative humidity of the test atmosphere shall be recorded.

(c) *Temperature of Specimen*.—Since

the mechanical properties of many plastics change rapidly with small changes in temperature, and since heat is generated as a result of the rapid flexing of the specimen in fatigue, the air velocity over the specimen shall be known. In order to insure uniformity of test conditions all tests shall be conducted in still air (average air velocity less than 50 ft. per min.). The temperature at the high stress region of the specimen shall be measured and recorded for a specimen operating at the fatigue strength in still air. This temperature shall be measured by means of a thermocouple attached to the specimen by a strip of cellulose tape  $\frac{1}{8}$  in. in width.

### Procedure

7. (a) *Calibration of Dynamometer.*—The dynamometer shall be calibrated occasionally by using a dummy specimen disconnecting the connecting rod at *F*, Fig. 3, hanging known weights from the wrist pin, *F*, and noting the corresponding reading of the dial, *H*. The load in pounds shall then be plotted against the corresponding dial reading. A straight line diagram should result. The load corresponding to one division of the dial may then be calculated from the slope of the line. This value is the calibration constant of the dynamometer. The calibration constant shall give results within 1 per cent of the correct value of the load.<sup>6</sup> The dynamometer dial, *H*, shall read zero when a specimen, without grip, *J*, attached, is fastened to the dynamometer at *M*.

(b) *Measurements.*—The minimum section of the specimen shall be measured to the nearest 0.001 in.<sup>6</sup> (For thin sheet, the relative error of measurement of the thickness of the sheet shall not exceed  $\frac{1}{3}$  of 1 per cent.) The specimen

shall be clamped snugly in the grips, *J*, Fig. 2, and the distance, *l*, from the center of the crankpin to the minimum section of the specimen shall be measured to the nearest 0.02 in.<sup>6</sup> The other end of the specimen shall be clamped in the vise and the connecting rod affixed to the wrist pin.

(c) *Calculation of Stress.*—The bending moment shall be calculated as follows:

$$M = S \frac{I}{c}$$

where:

*M* = bending moment,

*S* = desired stress in tension or compression, in pounds per square inch, on the outer (most highly stressed) fiber of the specimen,

*c* = distance in inches from the neutral surface to the outer fiber (Note), and

*I* = second moment (moment of inertia) of the net area at the minimum section calculated as follows:

$$I = \frac{bh^3}{12}$$

For the unnotched specimen (Fig. 4 (a)):

*b* = net width of the specimen, and

*h* = net depth of the minimum section of the specimen.

For the standard V-notched specimen (Fig. 4 (b)):

*b* = net width of the specimen, and

*h* = thickness of the specimen at the root of the notch.

For the drilled-hole notched specimen (Fig. 4 (c)):

*b* = width of the specimen minus the diameter of the hole, and

*h* = depth of the specimen.

NOTE.—For a rectangular or square cross-section (Figs. 4 (a) or (c)), *C* = one half the depth of the beam; for the notched specimen (Fig. 4 (b)), *C* = one half the thickness of the specimen at the root of the notch.

(d) *Calculation of Test Load.*—The load to be applied to the specimen shall then be calculated as follows:

$$P = \frac{M}{l}$$

<sup>6</sup> The maximum allowable errors indicated for the measurements and calibration will insure a maximum relative error, in the calculated stress, of less than 3 per cent.

where:

$P$  = load in pounds to be applied to the specimen,

$M$  = bending moment, and

$l$  = distance in inches from the wrist pin to the minimum section of the specimen.

The reading of the dynamometer dial corresponding to this load shall be determined from the dynamometer constant (Paragraph (a)).

(e) *Speed of Testing*.—The test shall be made at  $1720 \pm 25$  cycles of stress per minute, except when it is desired to determine the effect of speed of testing. The speed of testing shall be recorded.

(f) *Adjustments of Machine*.—To produce complete reversal of stress, the position of the vise shall first be adjusted so that the specimen is undeflected when the variable-throw crank is at mid-stroke. The variable-throw crank shall then be adjusted to produce the load desired on the specimen as indicated by the reading of the dynamometer dial. The load indicated shall be the same for both upward and downward deflection and shall be equal to that calculated in Paragraph (c) for the desired stress.

To produce an alternating cycle of stress in which the mean stress is different from zero, the variable-throw crank shall first be adjusted to produce the desired amplitude of the alternating cycle of stress and then with the variable-throw crank at mid-stroke the position of the vise shall be adjusted so as to produce a stress equal to the desired mean stress of the cycle.

(g) *Readings*.—Readings of the dynamometer and revolution counter shall be made before starting the machine. Dynamometer readings shall be recorded every 12 to 24 hr. during the testing of the specimen. After fracture of each specimen the cross-section at fracture and the distance from the fracture to the wrist pin shall be measured and recorded

before the specimen is removed from the grips. If the specimen fractures at a section more than  $\frac{1}{4}$  in. from the minimum section, the data for this specimen shall be marked void.

### Plotting and Interpreting Results

8. (a) No corrections for the following shall be made to the value of stress calculated at the minimum section of the specimen: (1) For fracture away from the minimum section, (2) for the position of the theoretical maximum stress (See Note, Section 4 (a)), or (3) for a change in dynamometer reading during the test.

(b) *Plotting Results*.—An S-N or stress-cycle diagram shall be plotted with the stress as the ordinate against the logarithm of the number of cycles required for fracture as the abscissa. The use of semilogarithmic paper will facilitate the plotting. Specimens which did not fracture shall be indicated on the diagram by an arrow directed away from the plotted point in the direction of increasing cycles.

(c) *Interpretation of Results*.—When a material shows a knee in the S-N or stress-cycle diagram such that the curve gives clear indications that it becomes asymptotic to a horizontal (constant stress) line, it is sufficient to carry the number of cycles far enough beyond the knee to indicate with a good degree of accuracy that the curve becomes asymptotic to a constant stress line. If the curve does not become asymptotic to a constant stress line, the test shall be continued until the number of cycles reached is greater than the number of cycles that the material will be expected to withstand in its life. The value of stress corresponding to this number of cycles shall be reported as the "fatigue strength at --- cycles." (Substitute the maximum number of cycles for which the stress-cycle diagram has been well defined by the tests.)

### Precautions Necessary in Applying Results

9. (a) So little is known about the relationship between service and fatigue tests of plastics that generous factors of safety should be used in design.

(b) It is necessary to make sure whether it is failure from fatigue that is the prime danger or failure from other causes such as creep, yielding, fracture under a single loading, or others.

(c) For plastics which are subject to creep at the temperature of testing (this probably includes all plastics at room temperature) a fatigue test, in which the mean stress is not zero, is accompanied by either creep or relaxation depending on the type of testing machine used. Relaxation of stress will occur for the type of machine considered in this method. As a result of this relaxation the range of stress tends to approach the condition of complete reversal of stress as time goes on. The magnitude of the effect that relaxation would have on the result obtained will depend upon the rate at which relaxation takes place and upon the time required for the endurance limit to be reached. The rate at which relaxation takes place will depend upon the material and the magnitude of the mean stress of the cycle.

### Report

10. The report (see Appendix II) shall include the following:

(1) Description of the material tested including name, manufacturer, code number, date of manufacture, type of molding, and thickness of specimen or original material in case of thin sheet,

(2) Dates of the test,

(3) Type of testing machine,

(4) Size and type of specimen used and whether notched or unnotched. If notched, the type of notch,

(5) Thickness of the specimen in the case of thin sheets,

(6) Preconditioning used (number of hours at the temperature in degrees Fahrenheit and relative humidity),

(7) Temperature of the testing room in degrees Fahrenheit and the relative humidity of the testing room,

(8) Temperature of the specimen in degrees Fahrenheit when operating at the fatigue strength,

(9) Speed (number of cycles per minute) of the testing machine,

(10) Fatigue ratio,

(11) Maximum number of cycles used in the test,

(12) Fatigue strength in pounds per square inch at the number of cycles of stress, and the mean stress, and

(13) Mean stress of the cycle in pounds per square inch and whether in tension or compression.

## APPENDIX I

### CLASSIFICATION OF FATIGUE TESTING MACHINES

I. *Direct Stress* (tension or compression, or both):

A. Repeated axial loading by alternating magnetic field or by inertia vibration.

B. Repeated axial deformation by means of connecting rod or cam.

II. *Flexural Stress*:

A. Repeated loading by:

(1) Rotating cantilever loaded by weight or spring.

(2) Fixed cantilever loaded by

magnetic or inertia vibrator or by rotating spring.

(3) Rotating beam in pure bending loaded by spring or weights.

B. Repeated deflection by:

(1) Fixed cantilever, repeated constant-deflection.

III. *Torsional Stress*:

A. Repeated angular torque by:

(1) Inertia vibrator.

B. Repeated angular twist by:

(1) Cam or connecting rod action on a torque arm.

## APPENDIX II

## SUGGESTED FORM OF DATA SHEET FOR REPEATED FLEXURAL FATIGUE TEST OF PLASTICS

## FATIGUE OF PLASTICS

Material..... Date.....  
 Fixed-cantilever, repeated-deflection type fatigue Test made by.....  
 testing machine No.....  
 Specimen:  
 Width,  $b$ ..... in. Initial weight..... g.  
 Height,  $h$ ..... in. Final weight..... g.  
 $\frac{I}{c} = \frac{bh^3}{6}$ ..... in.<sup>3</sup> Specimen temperature..... deg. Fahr.  
 Room temperature..... deg. Fahr. Room relative humidity..... per cent  
 Speed of testing..... cycles per minute  
 Amplitude of alternating stress in cycle..... psi. Mean stress of cycle..... psi.  
 Moment at minimum section..... in. lb. Arm to minimum section,  $l$ ..... in.  
 Load..... lb., or..... division on dynamometer  
 Cross-section of specimen at fracture:  $b$ ..... in.,  $h$ ..... in.  
 Distance center line of connecting rod pin to fracture,  $l$ ..... in.  
 Final reading revolution counter.....  
 Initial reading revolution counter.....  
 Cycles for fracture.....  
 Remarks:.....

## APPENDIX III

## SUGGESTED FORM FOR REPORTING RESULTS OF REPEATED FLEXURAL FATIGUE TEST OF PLASTICS

- For evaluation of the fatigue strength of a material: The report shall contain the following information:
  - Fatigue strength at ..... cycles of stress was ..... psi. for a mean stress of ..... psi. tension or compression (cross out one).
  - Fatigue ratio was .....
  - Maximum number of cycles used in the test was ..... cycles.
  - Temperature of the testing room was ..... deg. Fahr.
  - Relative humidity of the testing room was ..... per cent.
  - Temperature of the specimen when operating at the fatigue strength was ..... deg. Fahr.
  - Conditioning used was ..... hr. at ..... deg. Fahr. and ..... per cent relative humidity.
  - The type of testing machine was .....
  - The speed (number of cycles per minute) of the testing machine was .....
  - Description of material tested is: name ....., manufacturer ....., code number ....., date of manufacture ....., type of molding ....., thickness of sample .....
  - The dates of test were .....
- For evaluation of the fatigue strength of a notched material: The report shall contain items 1(b) through (k) above, and:
 

Fatigue strength at ..... cycles of stress of a standard V-notch specimen was ..... psi. for a mean stress of ..... psi. tension or compression (cross out one).

For evaluation of the fatigue strength of thin sheet<sup>5</sup>: The report shall contain items 1(b) through (k) above, and:

Fatigue strength at ..... cycles of stress of a sheet of ..... in. thickness was ..... psi. for a mean stress of ..... psi. tension or compression (cross out one).
- For evaluation of the fatigue strength of notched thin sheet: The report shall contain items 1(b) through (k) above, and:
 

Fatigue strength at ..... cycles of stress of a sheet of ..... in. thickness with a drilled-hole notch was ..... psi. for mean stress of ..... psi. tension or compression (cross out one).

# *Tentative Method of*

## FLEXURAL TEST OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 790 - 45 T

ISSUED, 1944; REVISED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers the procedure for determining the flexural properties of rigid plastic and electrical insulating materials, including molded thermoplastics and thermosetting materials and laminated plastics. This method is not applicable to nonrigid plastic materials.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Testing Machine*.—A properly calibrated standard type testing machine which can be operated at a constant rate of crosshead movement, and in which the error for indicated loads which are to be recorded shall not exceed plus or minus 1 per cent. The stiffness of the testing machine shall be such that the total elastic deformation of the system does not exceed 1 per cent of the total deflection of the test specimen. The load indicating mechanism shall be essentially free from the inertia lag at specified rate of testing.

The accuracy of the testing machine shall be verified in accordance with the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4).<sup>3</sup>

(b) *Loading Nose*.—The loading nose shall have a radius of at least  $\frac{1}{8}$  in. If significant indentation occurs, a radius of one and one-half times the specimen depth for a chord length of not less than twice the specimen depth shall be used.

(c) *Supports*.—Center loading shall be used. The span shall be adjustable. Either round-nosed supports of the same radius as the loading nose or roller-bearing plates shall be used. The roller-bearing plate is illustrated in Fig. 1.

### Test Specimens

3. The specimen may be machined from sheets, plates, or molded shapes or may be molded to the desired finished dimensions.

(a) *Sheet Materials*.—For flatwise tests of sheet materials, the depth of the specimen shall be the thickness of the material. It shall be tested as a simple beam (load applied at the center and beam supported at each end of span). The

<sup>1</sup> Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



span shall be at least 16 times the depth and the length of the specimen shall be the span length plus at least 2 in. to permit an overhang of at least 1 in. on each end. The width of the specimen shall be at least  $\frac{1}{2}$  in. and shall be kept uniform, but the width shall be at least equal to the depth. For specimen tested edge-wise the thickness of the sheet shall be the width of the specimen, and the depth shall not exceed the width. The span-depth ratio shall be at least 16 based on the nominal thickness (depth) (Notes 1, 2, 3, and 4).

NOTE 1.—When the width is more than one eighth of the span the beam formula will not strictly apply because the behavior of specimen becomes more nearly that of a plate than a beam. Hence, whenever practicable the width should be limited to one eighth of the span.

NOTE 2.—The dimensions suggested for test specimens are shown in Table I.

NOTE 3.—The original surface finish shall not be disturbed on the top and bottom surfaces, except as noted hereafter. For sheets thicker than 2 in. or for thinner sheets where the testing machine will not accommodate the span for full depth, specimens  $\frac{1}{2}$  in. in thickness may be cut from the sheet. In such a case both upper and lower surfaces shall be machined and the location of the specimens with reference to the total thickness shall be noted in the report. The

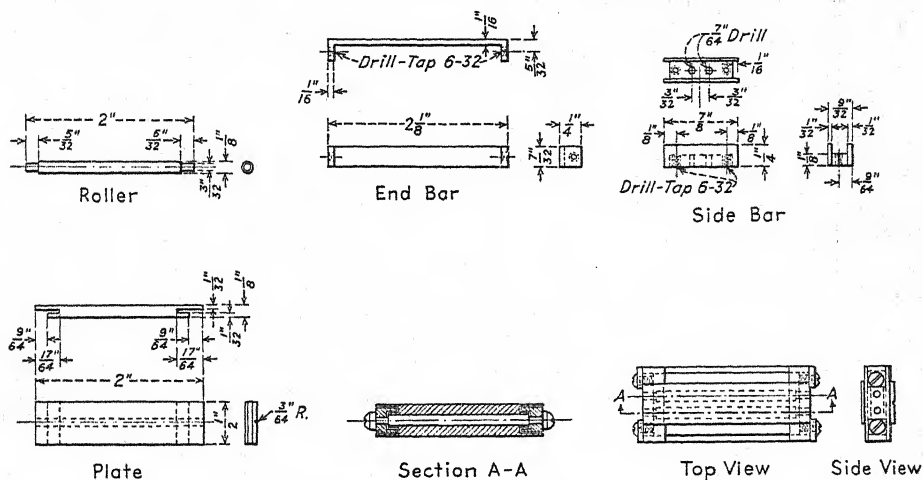


FIG. 1.—Roller-Bearing Plate for Supporting Specimen.

TABLE I.—SUGGESTED DIMENSIONS FOR TEST SPECIMENS.

Nominal Thickness of Sheet, in.	Span, min., in.	Flatwise		Edgewise	
		Depth of Specimen, in.	Width of Specimen, in.	Depth of Specimen, in.	Width of Specimen (Nominal Thickness), in.
		Min.	Max.		
$\frac{1}{16}$ .....	1	$\frac{1}{16}$	$\frac{1}{2}$	$\frac{1}{16}$	$\frac{1}{16}$
$\frac{1}{8}$ .....	1½	$\frac{1}{8}$	$\frac{1}{2}$	$\frac{1}{8}$	$\frac{1}{8}$
$\frac{3}{16}$ .....	2	$\frac{3}{16}$	$\frac{1}{2}$	$\frac{3}{16}$	$\frac{3}{16}$
$\frac{1}{4}$ .....	3	$\frac{1}{4}$	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{1}{4}$
$\frac{5}{16}$ .....	4	$\frac{5}{16}$	$\frac{1}{2}$	$\frac{5}{16}$	$\frac{5}{16}$
$\frac{3}{8}$ .....	6	$\frac{3}{8}$	$\frac{1}{2}$	$\frac{3}{8}$	$\frac{3}{8}$
$\frac{1}{2}$ .....	8	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$	$\frac{1}{2}$
$\frac{5}{8}$ .....	12	$\frac{5}{8}$	$\frac{1}{2}$	$\frac{5}{8}$	$\frac{5}{8}$
1.....	16	1	1	1	1
1½.....	24	1½	1½	1½	1½
2.....	32	2	2	2	2

values obtained on machined specimens and those with original surfaces retained are not strictly comparable, consequently any specification for flexural properties on the thicker sheets must state whether the original surfaces are to be retained or not.

NOTE 4.—For comparing values the thickness of specimens being compared should be approximately equal.

(b) *Molding Materials.*<sup>3a</sup>—For molding materials except as noted hereafter, the molded specimen for the flexural test shall be 5 by 0.5 by 0.25 in. using a span of 4 in. This provides a span-depth ratio of 16.

<sup>3a</sup> Editorially revised in June, 1946.

### Number of Test Specimens

4. (a) At least five specimens shall be tested for each sample in the case of isotropic materials.

(b) Ten specimens, five normal to, and five parallel with the principal axis of anisotropy, shall be tested for each sample in case of anisotropic materials.

(c) For sheet materials the specimens shall be tested as follows:

- (1) Five specimens cut lengthwise of the sheet, tested flatwise,
- (2) Five specimens cut lengthwise of the sheet, tested edgewise,
- (3) Five specimens cut crosswise of the sheet, tested flatwise, and
- (4) Five specimens cut crosswise of the sheet, tested edgewise.

(d) Specimens that break at some obvious fortuitous flaw shall be discarded and retests made, unless such flaws constitute a variable the effect of which it is desired to study.

### Conditioning Test Specimens

5. All test specimens prior to testing shall be conditioned in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

### Procedure

6. (a) The test shall be made in an atmosphere maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity. The width and thickness of the specimen shall be measured to the nearest 0.001 in. at the center of the span. The span length shall be measured to the nearest 0.01 in. The specimen shall be tested as a simple beam loaded at mid-span.

(b) *Speed of Testing.*—The rate of crosshead motion for testing flexural

specimens shall be calculated in accordance with the following formula:

$$N = \frac{ZL^2}{6d}$$

where:

$N$  = rate of crosshead motion in inches per minute,

$L$  = span in inches,

$d$  = depth of beam in inches,

$Z$  = unit rate of fiber strain in inches per inch of outer fiber length per minute, which constant shall be 0.01. This constant is the approximate calculated rate of fiber strain for a rate of crosshead motion of 0.05 in. per min., a span length of 4 in. and a depth of 0.5 in. such as have been used with the 5 by 0.5 by 0.5 in. test specimen.

The rate of crosshead motion shall be as near as possible but shall not exceed the value calculated from this formula. This permits the use of a constant rate of stressing provided the resultant rate of crosshead motion does not exceed the value calculated from the above formula.

(c) Load-deflection curves may be plotted to determine the modulus of elasticity, offset yield strength, and the total work measured by the area under the load-deflection curve. The deflection shall be measured with a dial gage or other suitable apparatus located under the specimen in contact with it at the center of the span, or by measuring the deflection of the neutral axis of the specimen. Data obtained by measuring the head travel are not acceptable.

(d) When load-deflection data are being taken, the rate of crosshead movement shall be low enough so that deflection gage readings can be made accurately.

### Retests

7. Results that deviate from the mean value of all tests should be rejected if the deviation of the doubtful value is more than five times the average deviation from the mean obtained by excluding the doubtful value. Such doubtful values shall be discarded and retests made, unless the degree of variability is a factor that is being studied.

### Calculations

8. (a) *Maximum Fiber Stress*.—The maximum fiber stress of the material tested as a simple beam of rectangular cross-section loaded at mid-span shall be calculated as follows:

$$S = \frac{3PL}{2bd^3}$$

where:

$S$  = maximum fiber stress in pounds per square inch,

$P$  = breaking load in pounds,

$L$  = distance between supports in inches,

$b$  = width of beam tested in inches, and

$d$  = depth of beam tested in inches.

(b) *Modulus of Rupture (Flexural Strength)*.—The modulus of rupture or flexural strength shall be calculated from the load sustained by the specimen at the moment of break, using the formula for maximum fiber stress given in Paragraph (a). This formula applies strictly only for materials for which the maximum fiber stress remains linearly proportional to strain up to the point of rupture. Since stress is not proportional to strain all the way to rupture, the modulus of rupture calculated as above will always be greater than the maximum fiber stress sustained by the specimen.

NOTE.—Some ductile materials will not break within the practical limit of deflection during the flexural test. With such materials, the maximum load sustained during a test shall be used in calculating the modulus of rupture.

### (c) *Flexural Offset Yield Strength*.—

The flexural offset yield strength is the stress at which the stress-strain diagram for the material departs from the initial direction of the curve by a specified increment of strain. For materials whose stress-strain diagram in the region of the yield is a smooth curve of gradual curvature, the offset method described in the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6)<sup>3</sup> may be conveniently used.

(d) *Modulus of Elasticity*.—The modulus of elasticity of material tested as a simple beam of rectangular cross-section loaded at mid-span shall be calculated as follows:

$$E_B = \frac{L^3}{4bd^3} \left( \frac{P}{Y} \right)$$

where:

$E_B$  = modulus of elasticity in bending in pounds per square inch,

$L$  = distance between points of support in inches,

$b$  = width of beam tested in inches,

$d$  = depth of beam tested in inches, and

$\frac{P}{Y}$  = slope of initial straight line portion of load-deformation curve in pounds per inch of deflection.

(e) For each series of tests, the arithmetic mean of all values obtained shall be calculated to three significant figures and reported as the "average value" for the particular property in question.

(f) The deviation of each value from the "average value" shall be calculated and the arithmetic mean of these deviations determined. This arithmetic mean shall be reported to two significant figures as the "average deviation" of the particular series of results.

**Report**

9. The report shall include the following:

(1) Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, and previous history,

(2) Direction of cutting and loading specimens,

(3) Conditioning procedure,

(4) Depth and width of specimen,

(5) Span length,

(6) Span-depth ratio,

(7) Radius of supports and nose,

(8) Rate of crosshead motion in inches per minute calculated in accordance with Section 6 (b),

(9) Maximum deflection at center of span in inches or millimeters,

(10) Modulus of rupture calculated in accordance with Section 8 (b), average value and average deviation, and

(11) Modulus of elasticity in bending in pounds per square inch, calculated in accordance with Section 8 (d), average value and average deviation.

# *Tentative Method of Test for*

## MEASURING THE FLOW PROPERTIES OF THERMOPLASTIC MOLDING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 569 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers procedures for determining the following:

*Method A.*—The temperature at which a thermoplastic material attains a defined degree of flow when subjected to a prescribed pressure for a prescribed time in a specified extrusion mold, or

*Method B.*—The degree of flow which a thermoplastic attains when subjected to a prescribed pressure and temperature for a prescribed time in a specified extrusion mold.

The equilibrium state of the material in this test is not necessarily representative of the state at which the material is molded commercially.

### Apparatus

2. The apparatus for the flow test (see Fig. 1) shall be a constant-force, vertical-orifice type machine<sup>3</sup> consisting essentially of the following:

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Prior to its present publication as tentative, this method was published as tentative from 1940 to 1943, being revised in 1941. It was adopted in 1943, published as standard from 1943 to 1944, being revised in 1944 and republished as tentative.

<sup>3</sup> Rossi-Peakes flow tester as described in U. S. Patent 2,066,016.

(a) *Orifice.*—A vertical orifice  $\frac{1}{8}$  in. in diameter and  $1\frac{1}{2}$  in. in length, Fig. 1, 1, into which the material flows. The orifice is machined into a split cone 1 in. in diameter at the base which is clamped into a steam-heated block, 3. A thermometer well  $\frac{3}{16}$  in. in diameter and  $1\frac{1}{4}$  in. in depth is drilled into the split cone. Temperature readings are taken at this point.

(b) *Thermometer.*—A  $1\frac{1}{4}$ -in. immersion mercury thermometer having a diameter just under  $\frac{3}{16}$  in. and a temperature scale of not more than 20 C. per inch of length.

(c) *Charge Chamber.*—Below the orifice and concentric with it is the charge chamber, 2,  $\frac{3}{8}$  in. in diameter and  $\frac{3}{4}$  in. in length.

(d) *Block.*—A steam-heated block, 3. Heat is supplied by steam at a line pressure of 90 to 150 psi., passing through an accurate reducing valve; if the line pressure fluctuates too widely, two reducing valves in series may be used. The temperature is controlled by regulating the steam pressure.

(e) *Ram.*—A steam-heated ram, 4, so

arranged that it applies pressure to the charge chamber from the bottom, forcing the material into the orifice. The steam line is so arranged that the steam passes through the reducing valve, the ram, the block, and finally through a suitable trap or small vent.

(f) *Pressure System*.—A mechanical system, 5, for applying a net pressure of 1500 psi. to the ram. The pressure

material in the orifice. A flexible chain attached to the upper part of the follower rod is passed part way around and fastened to the small diameter of a two-step pulley having a 3:1 ratio and mounted on the swing arm. A second flexible chain passing part way around and fastened to the larger diameter is attached to a sliding indicator and a

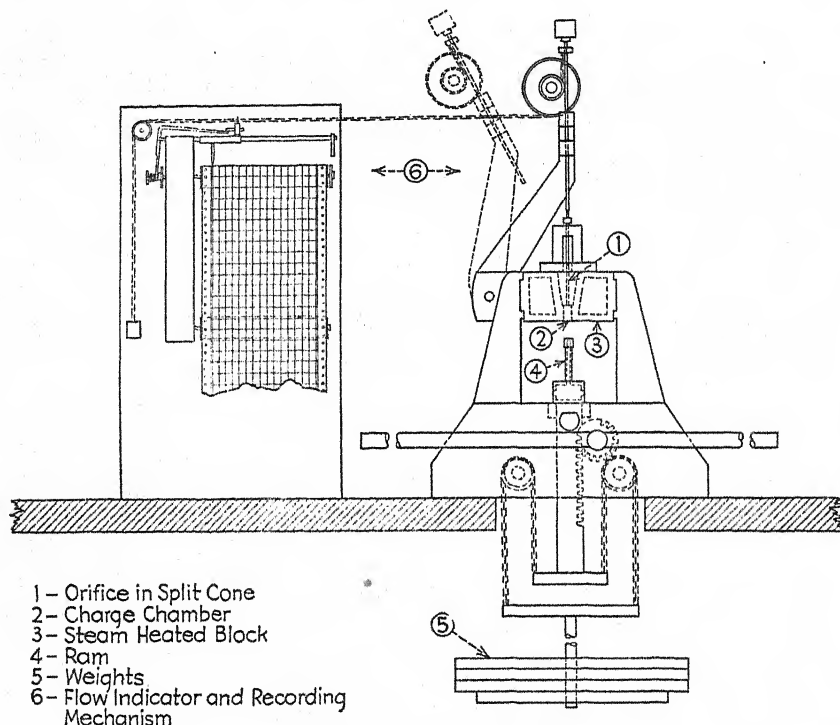


FIG. 1.—Flow Test Apparatus.

system illustrated in Fig. 1 is built so that any pressure up to 3,000 psi. may be applied in increments of 100 psi.

(g) *Flow Measuring Device*.—Means for measuring the flow of material into the orifice. Measurements shall be accurate to plus or minus 0.01 in. The following measuring systems are suitable:

(1) A follower rod, guided by loose bearings in a swing arm, rests on the

counterweight. The indicator travels over a calibrated scale so that the amount of flow in the orifice is shown on the scale, magnified three times. The weight of the flow-indicating mechanism is such that a pressure of 40 psi. is exerted on the material in the orifice; this however, is counterbalanced by sufficient additional weight on the pressure system, 5.

(2) *Optional*.—The flow measuring

device illustrated in Fig. 1 may be equipped with a time clock, a recording pen, and suitable graph paper with the scale as one axis and time as the other so that the flow behavior over any given time period may be recorded.

### Test Specimens

3. The test specimens shall be molded, machined, or preformed cylinders  $\frac{3}{8}$  in. in diameter and  $\frac{3}{8}$  in. in height. They shall be free of air bubbles and they shall be as free as possible of strains.

### Conditioning

4. (a) *Preferred Method.*—Test specimens shall be conditioned at room temperature in a desiccator over anhydrous calcium chloride for 72 hr.

(b) *Alternative Method.*—Test specimens shall be conditioned in an oven at 50 C. for 24 hr.

#### Method A

### Procedure<sup>4</sup>

5. (a) A working pressure of 1500 psi. shall be used.

(b) The unit of flow time shall be 2 min.  $\pm$  1 sec. as measured with a stop watch or timer.

(c) The test specimens at room temperature shall be inserted in the hot charge chamber and tested immediately.

(d) If poisoning of the orifice occurs, it shall be cleaned with acetone, benzene, or other suitable solvent before each test.

(e) Each material shall be tested at three or more temperatures at which the flow will be within the interval of 0.50 to 1.50 in. with at least one measurement above and one below 1 in. Temperatures shall be controlled within plus or minus 0.50 C. All temperature measurements shall be made at the split cone after it has come to equilibrium with the block (Note).

NOTE.—The split cone and the ram should be at the same temperature and both temperatures should be held as near constant to any chosen temperature as possible. In order to assure this, it is essential that the condensate be bled from the steam lines in such a manner that a minimum fluctuation of steam pressure is introduced. This may be accomplished by the use of an orifice rather than a trap in the efflux steam line. Approximate orifice sizes suggested are:

Temperature	Orifice Size, in.
100 to 120 C.....	$\frac{3}{16}$
120 to 140 C.....	$\frac{1}{4}$
140 to 160 C.....	$\frac{5}{16}$
160 to 180 C.....	$\frac{3}{4}$

(f) At the beginning of each series or at least once a day, the flow tester shall be checked against a sample of predetermined flow. At least two readings shall check within plus or minus 0.05 in. or plus or minus 5 per cent of the predetermined value, whichever is greater.

### Plotting Results

6. The linear flow shall be plotted against temperature on semilogarithmic paper. With the flow measurements plotted on the logarithmic coordinate and temperature on the linear coordinate, a straight line will usually result. The temperature at which the flow is exactly 1.0 in. shall be read from the graph and reported as the flow temperature.

### Report

7. The report shall include the following:

- (1) A statement indicating the nature of the material tested,
- (2) Curve of linear flow against temperature (Section 6),
- (3) Temperature at which flow is 1 in.,
- (4) Report of any unusual behavior of the test specimens such as discoloration, sticking, etc., and
- (5) Details of conditioning.

<sup>4</sup> Editorially revised, May, 1945.



*Method B***Procedure<sup>4</sup>**

8. (a) A working pressure of 500 psi., 1000 psi., or 1500 psi. shall be used.

NOTE.—In the case of polystyrene molding compositions, whenever possible, a temperature of 135 C. should be used with a pressure of either 1500 psi. or 500 psi., 1500 psi. being preferred.

(b) The unit of flow time shall be 2 min.  $\pm$  1 sec. as measured with a stop watch or timer.

(c) The temperature shall be held constant at some multiple of 5 C., for example 130, 135, 140, 145, 150, 155, 160, 165 C., etc., at which the flow will be within the interval of 0.50 to 1.50 in. Temperatures shall be controlled within plus or minus 0.5 C. All temperature measurements shall be made at the split cone after it has come to equilibrium within the block (see Note, Section 5(e)).

(d) The test specimen at room temperature shall be inserted in the hot charge chamber and tested immediately.

(e) All tests shall be run in duplicate.

(f) If poisoning of the orifice occurs, it shall be cleaned with acetone, benzene, or other suitable solvent before each test.

(g) At the beginning of each series of determinations or at least once a day, the flow tester shall be checked against a sample of predetermined flow. At least two readings shall check within plus or minus 0.05 in. or plus or minus 5 per cent of the predetermined value, whichever is greater.

**Report**

9. The report shall include the following information:

(1) Statement indicating the nature of the material tested,

(2) Temperature and pressure at which tests are run,

(3) Degree of flow to the nearest one hundredth of an inch,

(4) Report of any unusual behavior of the test specimen such as discoloration, sticking, etc.

(5) Details of conditioning.

## *Tentative Method of*

### DESIGNATING THE FLOW TEMPERATURE OF THERMO- PLASTIC MOLDING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 863 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. This method of flow designation is based on method A of the Tentative Method of Test for Measuring the Flow Properties of Thermoplastic Molding Materials (A.S.T.M. Designation: D 569)<sup>3</sup> and is applicable to cellulosic molding compounds and to molding compounds for which method A of A.S.T.M. Method D 569 is applicable.

#### Method

2. The flow temperature shall be de-

termined by method A of A.S.T.M. Method D 569.

#### Flow Designation

3. M flow shall be defined as  $293 \pm 9$  F. ( $145 \pm 5$  C.) and all other flows shall extend from this temperature in multiples of 9 F. (5 C.) as follows:

Flow Designation	Flow Temperature	
	Deg. Fahr.	Deg. Cent.
H <sub>n</sub> .....	for each 9 F. (5 C.) higher, add 1 flow (that is, H <sub>3</sub> , H <sub>4</sub> , H <sub>5</sub> , etc.)	
H <sub>2</sub> .....	320 $\pm$ 9	160 $\pm$ 5
H.....	311 $\pm$ 9	155 $\pm$ 5
MH.....	302 $\pm$ 9	150 $\pm$ 5
M.....	293 $\pm$ 9	145 $\pm$ 5
MS.....	284 $\pm$ 9	140 $\pm$ 5
S.....	275 $\pm$ 9	135 $\pm$ 5
S <sub>2</sub> .....	266 $\pm$ 9	130 $\pm$ 5
S <sub>n</sub> .....	for each 9 F. (5 C.) lower, add 1 flow (that is, S <sub>2</sub> , S <sub>4</sub> , S <sub>5</sub> , etc.)	

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Administrative Committee on Standards, January 17, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# *Tentative Method of Test for*

## ROCKWELL HARDNESS OF PLASTICS AND ELECTRICAL INSULATING MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 785 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method covers the procedure for testing the indentation hardness of plastics and electrical insulating materials by means of the Rockwell hardness tester.

(b) The results obtained by this method are a measure of the indentation produced by the penetrator described in the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18).<sup>3</sup> For certain types of materials having creep and recovery, the time factors involved in applications of major and minor loads have a considerable effect on the results of the measurements.

### Apparatus

2. The Rockwell hardness tester shall be used, conforming to the requirements of A.S.T.M. Methods E 18.<sup>3</sup> The flat anvil shall be used.

### Test Specimens

3. The test specimen shall have a minimum thickness of  $\frac{1}{4}$  in. unless it has been

verified that, for the thickness used, the hardness values are not affected by the supporting surface and that no imprint shows on the under surface of the specimen after testing. The specimen may be a piece cut from a molding or sheet or it may be composed of a pile up of several pieces of the same thickness, provided that precaution is taken that the surfaces of the pieces are in contact and not held apart by burrs from saw cuts, or other protrusions. Care shall be taken that the test specimen has parallel flat surfaces to insure good seating on the anvil and thus avoid the deflection which may be caused by its lack. The specimen shall be at least 1 in. square if cut from sheet stock, or at least 1 sq. in. in area if from other shapes. The minimum width shall be  $\frac{1}{2}$  in.

### Conditioning

4. Unless otherwise specified, specimens prior to testing shall be brought to a standard condition in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>4</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> 1946 Book of A S.T.M. Standards, Parts I-A and I-B.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## Procedure

5. (a) The procedure described in Sections 2 to 7 of A.S.T.M. Methods E 18<sup>3</sup> shall be followed.

(b) All tests shall be made at a temperature of  $25 \pm 2$  C. ( $77 \pm 3.6$  F.), unless otherwise specified.

(c) Unless otherwise specified in the individual methods or specifications for a particular material, either the M or R scale (Note 1) shall be used, the choice of scale to be determined as follows: If readings with the M scale show a total indentation of less than 150 scale divisions upon application of the major load, the M scale shall be used for the Rockwell test. If readings show a total indentation of 150 or more scale divisions the tester shall be changed to the R scale combination. The total indentation shall be determined by the number of divisions the pointer passes through during 7.5 sec. from the time of tripping the handle.

(d) The adjustment of speed of load application and the time of application of the major load are of great importance. The dash pot on the Rockwell tester shall be so adjusted that the operating handle completes its travel in from 4 to 5 sec. with no specimen on the machine and with the machine set up to apply a major load of 100 kg. When so adjusted the period taken for the mechanism to come to a stop with the specimen in place will vary from 5 to 15 sec. depending upon the particular specimen, the penetrator and load used.

(e) With the specimen in place, the minor load of 10 kg. shall be applied and the zero setting made within 10 sec.; the major load shall be applied immediately after the zero setting has been completed. In order to eliminate errors due to "creeping," and to the possibility of the weight arm descending far enough to rest on the lifting link or pin (Note 2) instead of on the specimen, the major load shall be removed from the specimen within 7 to 8 sec. after the handle has been tripped,

time being determined by a stopwatch. The Rockwell readings on the red scale shall be taken to the maximum nearest full scale division reached within 45 sec. after removing the major load.

(f) Care shall be taken that the test is not made so near the edge of the specimen that it will break out when the major load is applied. The specimen shall be supported in such a manner as to prevent errors due to overhang, when it does not balance itself on the anvil.

(g) The readings shall be recorded as follows: Count the number of times the needle passes through zero on the red scale on the application of the major load. Subtract from this the number of times the needle passes through zero upon the removal of this load. If the difference is zero, the reading shall be recorded as over 100. If the difference is 1, the reading shall be recorded as between 0 and 100, and, if the difference is 2, the reading shall be recorded as the scale reading -100 (Note 3).

NOTE 1.—The M scale requires a  $\frac{1}{4}$  in. ball penetrator, a minor load of 10 kg., a major load of 100 kg., and the reading taken on the red scale. The R scale requires a  $\frac{1}{2}$  in. ball penetrator, a minor load of 10 kg., a major load of 60 kg. and readings taken on the red scale.

NOTE 2.—If the arm rests on the link or pin, the load or the penetrator or both should be changed.

NOTE 3.—*Example:* With a difference of 2 and a scale reading of 97, indentation hardness values should be reported as  $-3 = (97-100)$ .

## Report

6. The report shall include the following:

(1) The Rockwell hardness number read on the red scale. The letter M or R shall precede the Rockwell hardness number depending on the combination which has been used in determining the hardness,

(2) The conditioning procedure used,

(3) The total thickness of the specimen, and

(4) The number of pieces in the specimen and their average thickness.

# *Tentative Method of Test for*

## HAZE OF TRANSPARENT PLASTICS BY PHOTOELECTRIC CELL<sup>1</sup>



A.S.T.M. Designation: D 672 - 45 T

ISSUED, 1942; REVISED, 1944, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is designed to measure photoelectrically the haze of colored or colorless transparent plastics, using specimens with substantially plane, parallel faces. It is not recommended for the measurement of low ranges of haze (less than 3 per cent), nor is it intended for use in evaluating translucent plastics, nor for measuring light transmission.

### Definition

2. *Haze*.—Haze is that fraction of the total transmitted light from a normally incident beam which is not transmitted in a straight line, and is calculated as follows:

$$\text{Haze, per cent} = \frac{T - T_r}{T} \times 100 = \frac{T_d}{T} \times 100$$

where:

$T$  = total light transmitted,

$T_r$  = amount of light transmitted in a straight line; and

$T_d$  = amount of light transmitted in all directions except rectilinearly.

### Apparatus

3. (a) The apparatus shall consist of a haze meter, the optical geometry of which shall be as shown in Fig. 1. The light source shall be a concentrated filament tungsten lamp which shall be operated at its rated voltage. The photoelectric cell shall be of the barrier layer type having a sensitive surface 1.5 in. in diameter and a spectral response which approximates that of the average human eye.<sup>3</sup> A collimated beam from this lamp shall be made to pass through a circular aperture 1 in. in diameter, which it shall completely fill, and to fall centrally on the sensitive surface of the photoelectric cell 18 in. from the aperture. The collimation of the beam shall be such that in the region between the aperture and the photoelectric cell the convergence or divergence of the edge of the beam shall not exceed 20 min. of arc (Note).

NOTE.—If this condition is satisfied, the diameter of the illuminated spot on the photoelectric cell will lie between 0.9 and 1.1 in.

(b) The photoelectric cell shall be connected to a meter having such characteristics that its readings are accurately proportional to the quantity of light falling on the surface of the specified

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> The Weston "Photronic" cells types I and III, if corrected by "Viscor" filters, are satisfactory.

photoelectric cell throughout the range of light intensity to be employed in these measurements. The equipment shall be located in a dark room or enclosure so arranged that no stray light reaches the photoelectric cell. The output of the lamp shall be maintained constant throughout the measurement of each specimen.

### Test Specimens

4. The test specimens shall be sufficiently large to cover completely the sensitive area of the photoelectric cell. The surfaces of the specimens shall be substantially flat and parallel. The thickness of the specimen shall be as received or for comparative purposes shall be  $0.125 \pm$

the undeviated portion of the light and also that part of the scattered light which is not deviated at angles greater than about 90 deg.

(b) The specimen shall then be placed in position *B* (Fig. 1) in front of the aperture and the meter reading recorded (Note).

NOTE.—The photoelectric cell still receives the undeviated portion of the light beam but it now collects only that small fraction of the scattered light which is confined to the small solid angles subtended by the sensitive surface of the photoelectric cell at points in aperture *B*.

(c) The difference between the photoelectric current when the specimen is at position *A* and when at position *B* shall be considered to be a measure of the light scattered by the specimen.

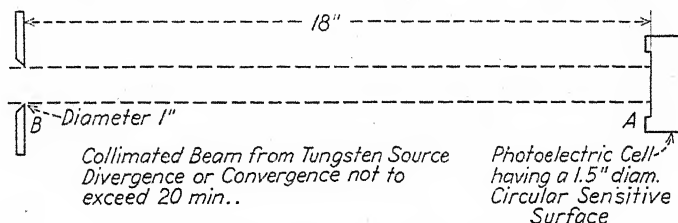


FIG. 1.—Geometry of Haze Meter.

0.005 in. Dust or grease shall be carefully removed without marring the surfaces.

### Procedure

5. (a) The test specimen shall be placed in position *A* (Fig. 1) directly in front of, parallel to, and in contact with the rim of, the photoelectric cell. The magnitude of the meter reading shall be adjusted by varying the current through the lamp within the rated voltage range or by use of a suitable shunt across the meter so that when the specimen is at position *A* the reading can be made to an accuracy of 0.1 per cent of the total (Note).

NOTE.—Of the light incident on the specimen, a fraction is transmitted undeviated and a fraction is scattered by the surfaces and the interior of the specimen. The photoelectric cell receives

(d) At least three measurements of haze shall be made and averaged for each specimen.

### Calculation

6. The haze expressed as a percentage shall be calculated as follows:

$$\text{Haze, per cent} = \frac{T_A - T_B}{T_A} \times 100$$

where:

$T_A$  = reading of meter with specimen at position *A*, and

$T_B$  = reading of meter with specimen at position *B*.

### Report

7. The report shall include the following:

- (1) Thickness of the test specimens, and
- (2) Average haze expressed as a percentage.

# Tentative Method of Test for HEAT DISTORTION TEMPERATURE OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 648 - 45 T

ISSUED, 1941; REVISED, 1944, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. (a) This method of test covers a procedure for determining the temperature at which an arbitrary deformation occurs when specimens are subjected to an arbitrary set of testing conditions. Data obtained by this method may be used to predict the behavior of plastic materials at elevated temperatures only in applications in which the factors of time, temperature, method of loading, and fiber stress are similar to those specified in this test. The test is particularly suited to control and development work.

(b) This method applies to molded and sheet materials available in thicknesses of  $\frac{1}{8}$  in. or greater and which are rigid at normal temperatures.

NOTE.—Sheet stock less than 0.125 in. but more than 0.040 in. in thickness<sup>2a</sup> may be tested by use of a composite sample having a minimum thickness of 0.125 in. The laminae must be of uniform width in order to obtain a uniform stress distribution. One type of composite specimen has been prepared by cementing the ends of the laminae together and then smoothing the edges with sandpaper. The direction of loading shall be perpendicular to the edges of the individual laminae.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the joint jurisdiction of the A.S.T.M. Committee D-9 on Electrical Insulating Materials and Committee D-20 on Plastics.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, May 10, 1945.

<sup>2a</sup> Investigations are under way to determine the minimum thickness of sheet stock that may be used in making a built-up specimen.

## Apparatus

2. The apparatus shall be constructed essentially as shown in Fig. 1 and shall consist of the following:

(a) *Specimen Supports*.—Metal supports for the specimen which shall be 4 in. apart, with the load applied on top of the specimen vertically and midway between the supports. The contact edges of the supports and of the piece by which pressure is applied shall be rounded to a radius of  $\frac{1}{8}$  in. The vertical members which attach the specimen supports to the upper plate shall be made of a material having the same coefficient of linear thermal expansion as is used for the rod through which the load is applied (Note 1).

NOTE 1.—Unless these parts have the same coefficient of linear expansion, the differential change in length of these parts introduces an error in the reading of the apparent deformation of the specimen. A test should be made on each apparatus using a test bar made of material having a low coefficient of expansion.<sup>3</sup> The temperature ranges to be used should be covered and a correction factor determined for each temperature. If this factor is 0.0005 in. or greater, its algebraic sign should be noted and the factor should be applied to each test by adding it algebraically to the reading of apparent deflection of the test specimen.

<sup>3</sup> Invar or Pyrex glass have been found suitable for this purpose.



(b) *Immersion Bath.*—A suitable liquid heat-transfer medium (Note 2) in which the specimen shall be immersed. It shall be well stirred during the test and shall be provided with a means of raising the temperature at  $2 \pm 0.2$  C. per min. (Note 3).

NOTE 2.—A liquid heat-transfer medium shall be chosen which will not affect the rigidity of the specimen. Mineral oil has been found suitable for most plastic materials.

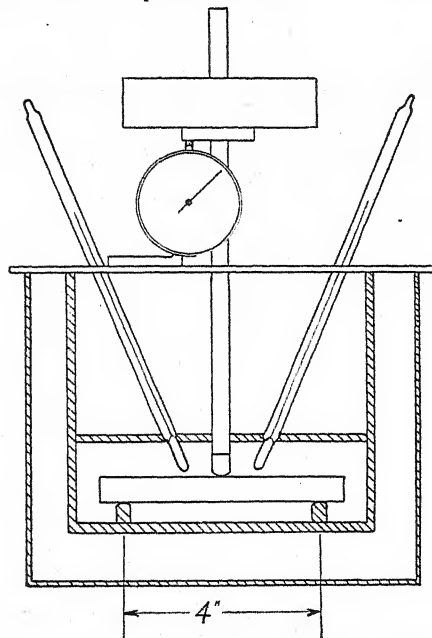


FIG. 1.—Apparatus for Heat-Distortion Test.

NOTE 3.—The specified heating rate can be conveniently maintained by the use of an electric hot plate or immersion heater, if the current through these units is regulated by a variable transformer or rheostat.

(c) *Weights.*—A set of weights of suitable sizes so that the specimen can be loaded to a fiber stress of 264 psi.  $\pm 2\frac{1}{2}$  per cent for the first part of the test described in Section 6 (a) and 66 psi.  $\pm 2\frac{1}{2}$  per cent for the procedure described in Section 6 (b). The weight of the rod which applies the testing force shall be determined and included as part of the total load. If a dial gage is used, the force exerted by its spring shall be determined and shall be included as part of

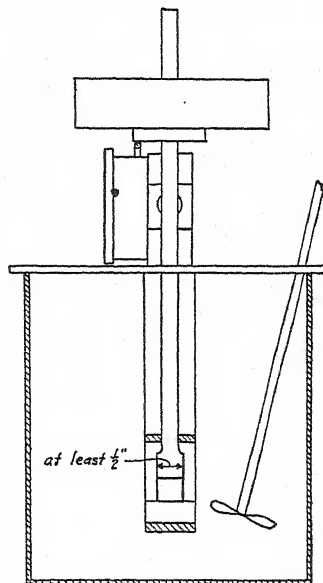
the load (Notes 4 and 5). The load shall be calculated from the following formula:

$$P = \frac{2Sbd^2}{3l}$$

where:

$P$  = load in pounds,

$S$  = maximum fiber stress in the specimen (264 psi. when tested according to Section 6 (a), 66 psi. when



tested according to Section 6 (b)),

$b$  = width of specimen in inches,

$d$  = depth of specimen in inches, and

$l$  = width of span between supports in inches.

NOTE 4.—In some designs of this apparatus the force of the dial gage spring is directed upward and must be subtracted from the load, while in other designs this force acts downward and must be added to the load.

NOTE 5.—Since the force exerted by the spring in certain dial gages varies considerable over the stroke, this force should be measured in that part of the stroke which is to be used.

(d) *Thermometers.*—The thermometer shall be one of the following, or its equiv-

alent, as specified in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).<sup>4</sup> thermometer 1C-39, 1F-39, 2C-39, or 2F-39, whichever temperature range is most suitable for material being tested.

#### Preparation of Apparatus

3. The apparatus shall be arranged so that the deflection of the mid-point of the specimen can be measured on a scale calibrated in five ten-thousandths of an inch. The apparatus may be arranged to shut off the heat automatically and sound an alarm when the specified deflection has been reached. Sufficient heat-transfer liquid shall be used to cover the thermometers to the point specified in their calibration, or 3 in. in the case of the A.S.T.M. thermometers referred to in Section 2(d)

#### Test Specimens

4. At least two test specimens shall be used to test each sample at each fiber stress. The specimens shall be 5 by  $\frac{1}{2}$  by any thickness from  $\frac{1}{8}$  to  $\frac{1}{2}$  in. (Note).

NOTE.—The types of mold and the molding process used to produce test specimens affect the results obtained in this test. Cooperating laboratories should therefore standardize mold and molding procedure to obtain concordant results.

#### Conditioning Test Specimens

5. The specimens shall be conditioned in accordance with the procedures specified for each material in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>4</sup>

#### Procedure

6. (a) The test specimen shall be placed in the apparatus with its  $\frac{1}{2}$ -in. dimension vertical. In the case of  $\frac{1}{2}$  by  $\frac{1}{2}$ -in. bars compression molded or cut from molded sheets, the bars shall be so placed that the direction of the testing force will be perpendicular to the direction of the molding pressure. The thermometers

shall extend to within  $\frac{1}{8}$  in. of the specimen but shall not touch it. The temperature of the bath shall be  $25 \pm 1$  C. at the start of each test, unless previous tests have shown that for the particular material under test no error is introduced by starting at a higher temperature. The load shall be adjusted so that the fiber stress is 264 psi.  $\pm 2\frac{1}{2}$  per cent as calculated by the formula given in Section 2 (c). The load shall be allowed to act for 5 min. (Note); the zero reading or setting of the measuring device shall then be made and the heating started. This waiting period may be omitted when testing materials which show no appreciable creep during the initial 5 min. The temperature at which the bar has deflected 0.010 in. shall be reported as the heat distortion temperature at 264 psi. fiber stress.

NOTE.—The 5-min. waiting period is provided to compensate partially for the creep exhibited by several materials at room temperature when subjected to the prescribed fiber stress. That part of the creep which occurs in the initial 5 min. is usually a large fraction of that which occurs in the first 30 min.

(b) The second part of the test shall be conducted exactly as prescribed in Paragraph (a), except that the load shall be adjusted to produce a fiber stress of 66 psi.  $\pm 2\frac{1}{2}$  per cent as calculated by the formula given in Section 2 (c). The temperature at which a deflection of 0.010 in. is reached shall be reported as the heat distortion temperature at 66 psi. fiber stress.

#### Report

7. The report shall include the following:

(1) The width and depth of the specimen measured to 0.001 in.,

(2) The heat distortion temperature for each fiber stress,

(3) The immersion medium and the average heating rate, and

(4) Any peculiar characteristics of the specimen noted during the test or after removal from the apparatus.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

*Tentative Method of Test for*  
LUMINOUS REFLECTANCE AND TRANSMISSION CHARACTERISTICS AND COLOR OF PLASTIC MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 791 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. (a) This method is intended as a basic, or referee test for determining luminous reflectance and transmission and color properties of substantially non-fluorescent plastics.<sup>3</sup> Less accurate means which are simpler, or more rapid, or make use of less expensive apparatus will be considered satisfactory to the degree that equivalent values are obtainable by the two methods.

(b) This method serves as a detailed supplement to the Standard Method of Test for Spectral Characteristics and Color of Objects and Materials (A.S.T.M. Designation: D 307)<sup>4</sup> which shall be considered as a part of this method to the extent that its instructions are applicable.

#### Apparatus

2. The apparatus shall conform to the requirements prescribed in Section 3 of A.S.T.M. Method D 307 with the exception of the following restrictions which shall be imposed upon the geometry of illuminating and viewing:

(a) The specimen shall be illuminated by a substantially unidirectional beam whose axis is normal to the specimen surface.<sup>5</sup>

(b) The maximum angle which any element of the beam makes with the direction of the axis, shall not exceed 3 deg.

(c) The beam shall be so regulated that its wave length spread in any portion of the visible spectrum shall not exceed 100 Å (Note 1).

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> Plastics, as specified in this method, shall include those materials implied in the scope of A.S.T.M. Committee D-20 on Plastics.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE 1.—Where spectral reflectance or transmission changes rapidly, it may be desirable to reduce the wave length spread until further re-

<sup>5</sup> For purposes of measuring total luminous reflectance, it may be necessary to tilt the specimen slightly. See Note 2 in Section 2.

duction ceases to affect the results. If this is done the sensitivity of the measuring instrument will be likewise reduced.

(d) The total light flux from the specimen (or, for reflection, the comparison standard), either in reflection or transmission, shall be collected and measured, such as in an integrating sphere. This collector shall preferably have an interior surface which is matte and highly reflecting throughout the visible range of wave length and shall have necessary holes not exceeding 3 per cent of the total surface (Note 2).

NOTE 2.—These requirements are met by a recording spectrophotometer of the Hardy type, except that, in some models, measurement of total luminous reflectance requires that glossy surfaces be tilted slightly to insure that the specularly reflected component of the beam is not lost in part or full through the entrance hole of the incoming beam. This may be accomplished by means of angle plates constructed as shown in Fig. 1, coated with a highly reflecting matte paint. To balance out possible errors, it is necessary to employ two, one for the specimen and one for the standard surface.

(e) The photometering device, whether visual or photoelectric, shall measure the integrated radiant flux by viewing, from a constant position, the brightness of a highly diffusing translucent window, such as opal glass, placed in a hole in the surface of the collector so as to become a smooth portion of that surface.

(f) The spectrophotometer shall be sufficiently accurate to warrant a precision of measurement of light flux which will give the reflectance or transmittance at any visible wave length within 1 per cent of the reading or within 0.001, whichever is larger.

(g) For measuring birefringent samples special auxiliary apparatus may be required in some cases (see Section 4 (c)).

### Test Specimens

3. (a) For absolute intercomparison, the surface of the specimens which are to

be measured shall be flat and, if the material is translucent, the rear surface shall also be flat and the two shall be substantially parallel. If the surface of the specimen which is to be measured is not flat, any results obtained shall be valid only for relative comparison with other specimens of the same shape and similarly positioned for measurement.

(b) For purposes of comparison of translucent materials and in the absence of good reason for using any other thickness, one or more of the following shall be chosen:

	Thickness, <sup>a</sup> in.
A.....	0.001
B.....	0.002
C.....	0.005
D.....	0.010
E.....	0.020
F.....	0.050
G.....	0.10
H.....	0.20
J.....	0.50
K.....	infinite

<sup>a</sup> All thicknesses are subject to a tolerance of plus or minus 10 per cent. Thickness K is defined in Section 2(i) of A.S.T.M. Method D 307.

NOTE.—For thick specimens which scatter light sensibly, that portion which would pass out through the edges must be considered. This may be done most readily by covering the edge with a substantially opaque layer of highly reflecting substance, such as white paint. When this procedure is found necessary, the dimensions of the specimen should be that of a right cylinder only sufficiently large to cover the measuring hole completely.

### Procedure

4. (a) Obtain spectral reflectance curves by comparing, at suitable wave length intervals, the total light reflected from the specimen, with that reflected from the standard surface.

NOTE 1.—Translucent materials should be backed with a dull black or other surface of equal absorbing power. For most accurate work a light trap is to be preferred.

(b) Obtain spectral transmission curves by comparing, at suitable wave length intervals, the total light incident upon the specimen, with that transmitted through it.

NOTE 2.—In the Hardy type spectrophotometer, comparison is made automatically and alternately by means of two beams, one of which serves for the standard and the other for the specimen. When measuring spectral transmission, the standard beam passes uninterrupted into the integrating sphere, while the sample beam passes first through the specimen. For most precise results the integrating sphere must be freshly smoked with magnesium oxide (see Note, Section 4 of A.S.T.M. Method D 307) at six-month intervals or more frequently, depending upon the general condition of the atmosphere. The two standard surfaces must be optically identical when used in measuring spectral transmission and, moreover, when

should preferably be measured in a non-polarizing instrument whose accuracy and precision has been shown, on isotropic plastics, to be equivalent to that obtained on an instrument meeting the requirements prescribed in Section 2.

As an alternative method use may be made of a specimen holder which rotates the specimen at double the speed of the second Rochon prism, either by direct gearing or by means of a second synchronous motor. A serious consideration of this method is the control of the

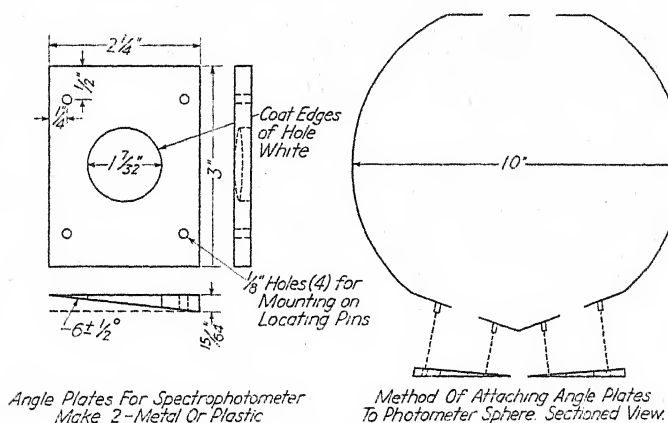


FIG. 1.—Angle Plates for Luminous Reflectance Test.

measuring specimens which scatter light, must be optically identical with the interior surface of the sphere.

(c) In some spectrophotometers the means for producing the standard and the sample beams so that their relative intensities may be readily and continuously varied to conform to the spectral characteristics of the specimen, requires that the beams be polarized. The most widely used of these instruments thus makes use of two plane polarized beams in which the axis of polarization is, however, rotating around the directional axis of the beam. This complicates the measurement, on this instrument, of plastics which exhibit birefringence and these

phase angle between the beam and the optical axis of the sample. Regulation of this angle will provide a family of spectral curves for the specimen, from the condition of maximum to that of minimum transmission, but will not provide a direct measure of the optical effect of the specimen in transmitting nonpolarized light.

A few spectrophotometric devices have been built in which the measuring beams are plane polarized, but do not rotate.<sup>6</sup> This removes the necessity for rotating the specimen.

<sup>6</sup> E. Stearns, "Application of Spectrophotometry in the Near Infrared," *Journal, Optical Soc. of Am.*, Vol. 33, No. 27, 1943.  
O. W. Pineo, U. S. Patent Nos. 2,107,836 and 2,126,410.

## Calculations

5. (a) The procedure described in A.S.T.M. Method D 307 shall be followed to obtain any or all of the values for luminous apparent reflectivity  $R_s$ , luminous apparent transmission  $T_s$ , and the trichromatic coefficients  $x$  and  $y$ , in terms of magnesium oxide as the standard of reflectance with the exception of the following two variations which shall be followed as necessary:

(1) If it is desired to determine the total luminous reflectance (including any specularly reflected component) of a specimen having a substantially glossy surface,  $R_s$  shall be calculated from the spectral reflectance curve obtained as indicated in Section 4.

(2) If it is desired to determine the luminous apparent reflectance of a glossy specimen, the value of spectral surface reflectance shall be calculated for normal incidence, based on the refractive index (Note 1) of the specimen, and subtracted at all wave lengths from the total luminous reflectance (Note 2).

NOTE 1.—The index of refraction shall be measured in accordance with the Standard Methods of Test for Index of Refraction of Transparent Organic Plastics (A.S.T.M. Designation: D 542),<sup>4</sup> and the reflection at the front interface then determined in air according to Fresnel's law as follows:

$$R_i = \left( \frac{n - 1}{n + 1} \right)^2$$

where:

$R_i$  = reflection on the front interface.

$n$  = index of refraction of the specimen.

NOTE 2.—The surface reflection will be substantially constant over the visible range of wave length. For  $n = 1.45$ ,  $R_i = 0.034$ ; for  $n = 1.70$ ,  $R_i = 0.067$  and linear interpolation will not introduce a significant error. In practice,

therefore, the total spectral reflectance curve may be obtained and  $R_s$  calculated; luminous apparent reflectance will then be equal to  $R_s - R_i$ .

(b) Eliminate the specular surface reflection of a glossy specimen in order to measure its color characteristics in accordance with the second variation of Paragraph (a). A new, uniformly depressed, curve will thus be obtained which is to be employed in calculating color by reflected light.

## Illuminant

6. Illuminants A, B, or C, as described in A.S.T.M. Method D 307, shall be mathematically included in the calculations of  $R_s$ ,  $T_s$ ,  $x$  and  $y$ , except that, in the absence of reason for other choice, illuminant C shall be preferably employed.

## Report

7. (a) The report of either or both the reflection and transmission characteristics of a specimen shall consist of the following:

(1) The total luminous reflectance or the luminous apparent reflectance as applicable, with the corresponding thickness,

(2) The luminous transmission, as applicable, with the corresponding thickness, and

(3) The designation of the standard illuminant employed.

(b) The report of the color characteristics of a specimen shall consist of the following:

(1) The trichromatic coefficients  $x$  and  $y$ , and the luminous transmission or luminous apparent reflectance  $Y$ ,

(2) The thickness of the specimen, and

(3) The designation of the standard illuminant employed.

# *Tentative Method of Test for*

## MEASURING RELATIVE MOBILITY OF THERMOSETTING MOLDING POWDER<sup>1</sup>



A.S.T.M. Designation: D 731-44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is designed to measure the mobility of thermosetting plastics under certain specified conditions of mold design and applied temperature and pressure. This method can be used to determine batch-to-batch uniformity of a given plastic, but cannot be used with certainty to determine whether different types of thermosetting materials are of the same mobility.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Mold*.—A cup mold of the flash type, as shown in Fig. 1, operated under applied constant pressure and temperature.

(b) *Thermometer*.—A  $1\frac{1}{4}$ -in. partial-immersion mercury thermometer having a diameter just under  $\frac{3}{16}$  in. and a temperature scale of not more than 20 C. per in. of length. A pyrometer may also be

used and the temperature of the mold surfaces determined.

(c) *Heating System*.—Any conventional means for heating the press platens provided the heat source is constant enough to maintain the molding temperature within plus or minus 1 C. of the specified temperature (Section 5 (b)).

(d) *Pressure System*.—A hydraulic pressure system regulated by a needle valve placed between the hydraulic supply and the pressure gage so that the ram shall travel at a speed of 5 in. in 4 to 5 sec. when the operating valve is completely open. The ram diameter of the press shall not exceed 4 in. The pressure gage shall have a dial range of not more than 5000 psi. and shall be accurate to within 1 per cent of the dial range. If a hydraulic pressure regulator is used to obtain the required line pressures, it shall be placed between the needle and the pressure gage.

(e) *Stop Watch*.

### Conditioning

3. (a) Material shall be tested in the as-received condition (Note 1), except

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.



in referee tests when it shall be conditioned in accordance with Paragraph (b).

NOTE 1.—Before samples in the as-received condition are tested, check tests should be conducted on samples conditioned in accordance with Paragraph (b) to determine whether the test systems are in agreement.

(b) For referee testing (Note 2), the material shall be spread to a depth of not more than  $\frac{1}{4}$  in. in a desiccator and conditioned for 72 hr. over anhydrous calcium chloride at room temperature. As an alternative procedure, the material shall be spread to a depth of not more than  $\frac{1}{4}$  in. and conditioned 16 to 18 hr. by drying at  $50 \pm 3$  C. ( $122 \pm 5.4$  F.),

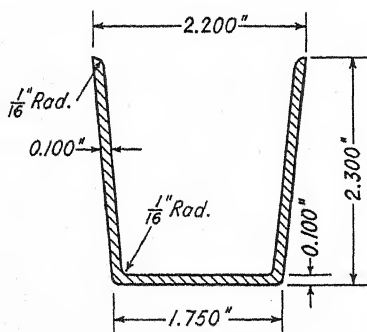


FIG. 1.—Cup Mold.

followed by cooling to room temperature in a desiccator.

NOTE 2.—The use of conditioned samples for referee testing is recommended to eliminate the moisture variable. The applicable method of conditioning materials for referee testing shall be agreed upon by the manufacturer and the purchaser.

### Test Sample

4. To determine the weight of the test sample a cup having a flash or fin thickness of not more than 0.006 in. shall be molded (Fig. 1). The adhering fin shall be removed and the cup weighed to the nearest 0.1 g. This weight multiplied by 1.1 shall be the weight of the test sample. The test sample may be in the form of loose powder or preformed sections.

### Procedure

5. (a) If the mold is not fixed in the press and a hand mold is used, the time required to remove the mold from the press, place the test sample in the mold cavity, replace the charged mold in the press, and apply the hydraulic pressure, shall be 5 to 10 sec. (Note 1).

NOTE 1.—The rate of flow is sensitive to the condition of the mold surfaces. Preceding materials may have deposited a film of lubricant over the mold surfaces or otherwise influenced the mold surfaces to the extent that erroneous results will be obtained unless the mold is properly conditioned prior to testing. The suggested procedure is to discard the first few cups molded and accept the flow time as correct when two successive cups molded under test do not differ by more than 1 sec. in time of flow.

(b) The preferred temperature of the mold shall be  $160 \pm 1$  C. ( $320 \pm 2$  F.) at the time the test sample is placed in the mold. Other temperatures may be used as agreed upon by the manufacturer and the purchaser.

(c) A load of 5000 lb. shall be used on the mold to make the initial test. If the cup is not completely filled, the test shall be repeated, increasing the load used by increments of 5000 lb. until a completely filled cup is obtained, and the closing time is not less than 10 sec. (Note 2). The corresponding gage points shall be determined by dividing the desired load by the area of the ram in square inches. The gage pressure shall be maintained constant within plus or minus 50 lb.

NOTE 2.—The preferred pressure to be used is that pressure which will result in a closing time of not less than 10 nor more than 40 sec. Some molding powders are formulated to give long, slow closing times with minimum molding pressures, and in such cases it is desirable to use pressures in excess of that required to completely fill the cup so as to obtain closing times of less than 40 sec.

(d) The time of flow shall be measured from the instant that the hydraulic gage shows an applied pressure to the instant that the test sample ceases to flow more than 0.001 in. per sec. (Note 3).

NOTE 3.—A convenient method for determining the final rate of flow is to indicate the final movement of the platens with a dial micrometer.

### Report<sup>3</sup>

6. The report shall include the following:

(1) A statement indicating the nature of the material tested and the manufacturer's material number and batch number,

(2) Time of flow in seconds to the nearest second,

(3) Total force in pounds used on the mold,

(4) Temperature of the mold,

(5) Weight of the test sample in grams, designated as loose powder or preform weight,

(6) Thickness of the bottom of the cup measured to the nearest 0.001 in. at its center,

(7) Report of any unusual characteristics of the test sample such as discoloration, sticking, etc., and

(8) Details of conditioning when the test sample is in other than the as-received condition.

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<sup>3</sup> Editorially revised, May, 1945.

# *Tentative Methods of Test for*

## RESISTANCE OF PLASTICS TO ACCELERATED SERVICE CONDITIONS<sup>1</sup>



A.S.T.M. Designation: D 756 - 46 T

ISSUED, 1944; REVISED, 1944, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These methods of test cover procedures for determining the weight and shape changes occurring in plastics under various conditions of use, not where exposure to direct sunlight, weathering, corrosive atmospheres or heat alone is involved, but where changes in atmospheric temperature and humidity are encountered. This embraces the interior of buildings, and the interior of transport facilities such as motor vehicles, airplane cargo spaces or wing interiors, holds of ships, and railroad cars. Procedures are provided for exposing plastics to combinations of extreme humidity and temperature that will accelerate the changes taking place in the materials kept in sheltered spaces but subject to humidity and temperature variation. (See Explanatory Note).

(b) Seven test procedures are provided which prescribe conditions for

different types of exposure. Six of the procedures cover exposures at graduated levels of temperature and extremes of humidity; the seventh prescribes conditions involving alternate exposure to high and low temperatures. Insofar as weight and shape changes, embraced under the general term of dimensional stability, are concerned, these procedures provide a method of test. Further use of any of the conditions set up in the procedures is suggested, such as conditioning schedules prior to physical testing of the plastic either to test it at the particular condition involved, or to study changes resulting from exposure to that condition.

### Significance of Tests

2. (a) The test conditions covered in these methods represent a start towards organizing a group of test procedures for determining the effects of specified changes of atmospheric temperature and humidity upon plastic articles. These procedures have been used for testing both thermosetting and thermoplastic materials.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

NOTE.—The test procedures covered in these methods have been drawn from various tests used in branches of the plastics industry, and by government agencies in procurement of materials.

(b) While several of the testing temperatures are not used in other A.S.T.M. methods, they have a background of several years experience in portions of the plastics industry. Especially in procedures IV and V (Sections 11 and 12) the accelerating effect of temperature in prompting changes caused by a rise in humidity leads to temperatures that are well above those encountered in normal service, and they are used simply to accelerate change.

(c) Procedure I (Section 8) has been found to develop warping, weight change, and exudation in plastic parts. Procedure II (Section 9) is designed to reveal poorly cured plastics by developing cracks in them. Procedures III and IV are more severe measures of the same tendencies developed in procedure I; the conditions of procedure IV will produce noticeable chemical decomposition in many plastics. Procedure V (Section 12) is especially valuable in testing the behavior of plastic parts with metallic inserts and laminates, for cracking on exposure to temperature change. Procedures VI and VII are modifications of procedure I, applying to impact-resistant and low heat-distortion temperature types of thermoplastics, respectively.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Balance*.—A balance capable of weighing accurately to 0.05 per cent a test specimen weighing 100 g. or less, and to 0.1 per cent a test specimen weighing over 100 g.

(b) *Oven*.—A circulation air oven capable of maintaining the required temperature of test within plus or minus 2 F.

(c) *Containers*.—Noncorroding containers with a shelf to support the test specimen above the solution used for maintaining the required humidity. The container shall be tightly sealed except for a small capillary which permits release of vapor pressure that might otherwise lift the top off the container. Each test specimen shall be tested preferably in a separate container.

(d) *Desiccator*.—A clean, dry, uncharged desiccator or equivalent closed container in which to bring test specimens to room temperature.

(e) *Absorbent Cloth*.—Clean, nonlinting absorbent cloth for use in wiping exudation or condensed moisture from test specimens.

(f) *Micrometer*.—A micrometer capable of measuring dimensions of test specimens to 0.001 in.

(g) *Cold Box*.—A cold box capable of maintaining the required temperature of test within plus or minus 5 F.

### Test Specimens

4. The general term plastics is used to describe the samples tested, since it is the intent of these methods to provide testing procedures which apply with equal validity to finished articles comprising plastics components together with other materials, to plastics parts, to plastics articles, to test specimens molded from plastics, and, if shape and nature of the material permit, to sheets, rods, tubes, or other plastic shapes furnished in a finally processed form. The shape, size, and process of forming greatly influence the behavior of plastic objects, hence a standard size test specimen is not prescribed in these methods, but the type of test specimen to be used shall be specified by the purchaser. Each test shall be made in duplicate.

### Conditioning

5. Unless otherwise specified, test specimens shall be conditioned prior to

testing and reconditioned at the end of the test procedure in accordance with the Standard Procedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup> Test specimens of material  $\frac{1}{4}$  in. or less in thickness of plastic section shall be conditioned for a minimum period of 40 hr., and specimens of material over  $\frac{1}{4}$  in. in thickness of plastic section shall be conditioned for a period of at least 88 hr.

#### Measurements of Test Specimens

6. (a) The following measurements shall be made on conditioned test specimens prior to testing, after reconditioning at the end of a test procedure, and at any intermediate stage as prescribed in the test procedures:

(1) *Weight*.—The weight within 0.05 per cent if the specimen weighs 100 g. or less, and within 0.1 per cent if the specimen exceeds 100 g. in weight.

(2) *Dimensions*.—The thickness to 0.001 in., the plane dimension in the direction of injection or transfer to 0.001 in., and the plane dimension across the direction of injection or transfer to 0.001 in.

(3) *Dimensions of Compression Molded Specimen*.—The thickness to 0.001 in., and the perpendicular dimensions in the plane at right angles to the direction of molding to 0.001 in.

(b) Specimens shall be brought to room temperature in the uncharged desiccator, which will require 10 to 30 min. Then the specimen shall be weighed in less than 10 min. after exposure to room conditions. The dimensions shall be measured immediately after weighing the specimen.

(c) At the discretion of the purchaser, requirements for weights and measurements at intermediate stages given in the procedures may be omitted.

#### Visual Examination

7. Noticeable qualitative changes in surface, outline, and general appearance of the test specimen shall be recorded after each stage of the testing procedure. These changes include color, surface irregularities, odor, and splits, in accordance with the Standard Descriptive Nomenclature of Objects Made from Plastics (A.S.T.M. Designation: D 675).<sup>3</sup> Changes shall also be noted as they occur, especially those which alter the shape so that intended dimensions are no longer significant.

#### Procedure I

8. (a) The test cycle for procedure I shall be as follows:

24 hr. at 140 F. and 88 per cent relative humidity, followed by  
24 hr. at 140 F. in the oven.

(b) Condition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(c) Expose the specimen for 24 hr. on the shelf of a container maintained at  $140 \pm 2$  F. in the oven, and containing a saturated solution of sodium sulfate to maintain a relative humidity of 85 to 89 per cent.

(d) Remove the specimen from the container, place it in the uncharged desiccator, and bring to room temperature in accordance with Section 6 (b).

(e) Wipe the specimen with the absorbent cloth, then weigh, measure dimensions, and examine visually in accordance with Sections 6 and 7.

(f) Within 2 hr. after completion of the operation prescribed in Paragraph (c), expose the specimen for 24 hr. in the oven at  $140 \pm 2$  F.

(g) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6 (b).

(h) Weigh the specimen, measure dimensions, and examine visually in accordance with Sections 6 and 7.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(i) Recondition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(j) The specimen may be subjected to physical tests in accordance with Section 15.

## Procedure II

9. (a) The test cycle for procedure II shall be as follows:

72 hr. at 140 F. in the oven.

(b) Weigh and measure dimensions of the specimen in the as-received condition in accordance with Section 6.

(c) Expose the specimen for 72 hr. in the oven at  $140 \pm 2$  F.

(d) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6(b).

(e) Weigh the specimen, measure dimensions, and examine visually in accordance with Sections 6 and 7.

## Procedure III

10. (a) The test cycle for procedure III shall be as follows:

24 hr. at 160 F. and 70 to 75 per cent relative humidity, followed by

24 hr. at 160 F. in the oven.

(b) Condition the specimen, weigh, and measure its dimensions in accordance with Sections 5 and 6.

(c) Expose the specimen for 24 hr. on the shelf of a container maintained at  $160 \pm 2$  F. in the oven, and containing a saturated solution of sodium chloride to maintain a relative humidity of 70 to 75 per cent.

(d) Remove the specimen from the container, place it in the uncharged desiccator, and bring to room temperature in accordance with Section 6(b).

(e) Wipe the specimen with the absorbent cloth, then weigh, measure dimensions, and examine visually in accordance with Sections 6 and 7.

(f) Within 2 hr. after the completion of the operation described in Paragraph (c), expose the specimen for 24 hr. in the oven at  $160 \pm 2$  F.

(g) Place the specimen in the uncharged desiccator and bring to room temperature in accordance with Section 6(b).

(h) Weigh the specimen, measure dimensions, and examine visually in accordance with Sections 6 and 7.

(i) Recondition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(j) The specimen may be subjected to physical tests in accordance with Section 15.

## Procedure IV

11. (a) The test cycle for procedure IV shall be as follows:

24 hr. at 175 F. over water, followed by

24 hr. at 175 F. in the oven.

(b) Condition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(c) Expose the specimen for 24 hr. on the shelf of a container maintained at  $175 \pm 2$  F. in the oven, and containing distilled water to maintain a humid atmosphere.

(d) Remove the specimen from the container, place it in the uncharged desiccator, and bring to room temperature in accordance with Section 6(b).

(e) Wipe the specimen with the absorbent cloth, then weigh, measure dimensions, and examine visually in accordance with Sections 6 and 7.

(f) Within 2 hr. after the completion of the operation described in Paragraph (c), expose the specimen for 24 hr. in the oven at  $175 \pm 2$  F.

(g) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6(b).

(h) Weigh the specimen, measure di-

mensions, and examine visually in accordance with Sections 6 and 7.

(i) Recondition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(j) The specimen may be subjected to physical tests in accordance with Section 15.

#### Procedure V

12. (a) The test cycle for procedure V shall be as follows:

- 24 hr. at 175 F. and 70 to 75 per cent relative humidity, followed by
- 24 hr. at  $-40 \pm 4$  F. or  $-70 \pm 4$  F., as specified,
- 24 hr. at 175 F. in the oven,
- 24 hr. at  $-40$  F. or  $-70$  F., as specified.

(b) Condition the specimen, weigh, and measure its dimensions in accordance with Sections 5 and 6.

(c) Expose the specimen for 24 hr. on the shelf of a container maintained at  $175 \pm 2$  F. in the oven and containing a saturated solution of sodium chloride.

(d) Remove the specimen from the container, and wipe excess moisture from it with the absorbent cloth.

(e) Within 30 min. after the completion of the operation described in Paragraph (c), expose the specimen for 24 hr. in a cold box maintained at  $-40 \pm 4$  F. or  $-70 \pm 4$  F., as specified.

(f) Place the specimen in the uncharged desiccator and bring to room temperature in accordance with Section 6 (b).

(g) Wipe the specimen with the absorbent cloth, then weigh, measure dimension, and examine visually in accordance with Sections 6 and 7.

(h) Within 2 hr. of the completion of the operation described in Paragraph (e), expose the specimen for 24 hr. in the oven at  $175 \pm 2$  F.

(i) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6 (b).

(j) Within 30 min. after the completion of the operation described in Paragraph

(h), expose the specimen for 24 hr. in the cold box at  $-40 \pm 4$  F. or  $-70 \pm 4$  F., as specified.

(k) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6 (b).

(l) Wipe the specimen with the absorbent cloth, then weigh, measure dimensions, and examine visually in accordance with Sections 6 and 7.

(m) Recondition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(n) The specimen may be subjected to physical tests in accordance with Section 15.

#### Procedure VI

13. (a) The test cycle for procedure VI shall be as follows:

- 24 hr. at 100 F. and 100 per cent relative humidity, followed by
- 24 hr. at 140 F. in an oven.

(b) Condition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(c) Expose the specimen for 24 hr. in a container maintained at  $100 \pm 2$  F. and 100 per cent relative humidity (obtained by using distilled water as the humidifying medium).

(d) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6 (b).

(e) Wipe the specimen with the absorbent cloth, then weigh, and measure dimensions and examine visually in accordance with Sections 6 and 7.

(f) Within 2 hr. after the completion of the operations described in Paragraphs (c) and (d), expose the specimen for 24 hr. in the circulation air oven at 140 F.

(g) Place the specimen in the uncharged desiccator and bring to room temperature in accordance with Section 6 (b).

(h) Weigh the specimen, measure dimensions, and examine visually in accordance with Sections 6 and 7.



(i) Recondition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(j) The specimen may be subjected to physical tests in accordance with Section 15.

## Procedure VII

14. (a) The test cycle for procedure VII shall be as follows:

24 hr. at 120 F. and 100 per cent relative humidity, followed by  
24 hr. at 120 F. in an oven.

(b) Condition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(c) Expose the specimen for 24 hr. in a container maintained at  $120 \pm 2$  F. and 100 per cent relative humidity (obtained by using distilled water as the humidifying medium).

(d) Place the specimen in the uncharged desiccator, and bring to room temperature in accordance with Section 6 (b).

(e) Wipe the specimen with the absorbent cloth, then weigh, and measure dimensions and examine visually in accordance with Sections 6 and 7.

(f) Within 2 hr. after the completion of the operations described in Paragraphs (c) and (d), expose the specimen for 24 hr. in the circulation air oven at 120 F.

(g) Place the specimen in the uncharged desiccator and bring to room temperature in accordance with Section 6 (b).

(h) Weigh the specimen, measure dimensions, and examine visually in accordance with Sections 6 and 7.

(i) Recondition the specimen, weigh, and measure dimensions in accordance with Sections 5 and 6.

(j) The specimen may be subjected to physical tests in accordance with Section 15.

## Physical Tests

15. On appropriate test specimens, provided that prohibitive changes in dimension, structure, or shape have not taken place, physical tests, such as impact resistance and flexural strength, may be conducted either at one of the test conditions, or after the final conditioning, for comparison with similar tests on specimens which have not been exposed to the conditions prescribed by these methods.

## Report

16. The report shall include the following:

(1) Description of test specimens, including material, shape, method of molding, and where obtained,

(2) Test procedures used, designated by Roman numerals and the number of cycles of each,

(3) Average values of percentage changes in weight and dimensions found after (a) each stage of the testing procedure, and (b) reconditioning, when the entire cycle is finished. Notation shall be made whether all measurements prescribed in Section 6 (a) were taken, or whether the simplified procedure of Section 6 (c) was used, and

(4) Description of qualitative changes in appearance that may have taken place during each stage of the test procedure.

## EXPLANATORY NOTE

NOTE.—To simulate the behavior of plastics subject to repeated exposure to heat and humidity, test procedures involving the use of repeated

cycles of dry heat and humid heat may be selected from the single cycle test procedures described in these methods. In making such

cycle tests, the initial conditioning shall be given, followed by exposure to the test as stated. Subsequent test procedures shall be applied without reconditioning the specimen at the end of each unit of the cycle. For instance, in carrying out a three-cycle exposure to procedure I, the first cycle shall be carried out in accordance with Sections 8 (a) to (h), the second cycle in accordance with Sections 8 (c) to (h),

and the third cycle in accordance with Sections 8 (c) to (j). The reconditioning shall always be carried out at the end of the test cycle unless sample failure makes this step appear trivial. In reporting such tests, mention should be made of the various stages selected for the cycle, and the number of cycles to which the specimen is subjected.

# *Tentative Methods of Test for* SPECIFIC GRAVITY OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 792-44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. (a) These methods of test are intended for the determination of the specific gravity of solid plastics. Two methods are covered, as follows:

*Method A.*—For plastics in a finished condition, whether machined or otherwise formed to the dimensions of a piece ready for service, such as sheets, rods, tubes, and molded articles.

*Method B.*—For plastics in preformed condition, such as molding powder or pellets, flake or the like.

(b) These methods are based on the displacement of a like volume of liquid and determination of the change in weight. The liquid used shall be water for plastics which are unaffected by water. For plastics which are soluble in or otherwise affected by water, kerosine or other suitable liquids may be used. The liquid should wet the plastic but should not otherwise affect it and should have a lower density than the plastic.

## Specific Gravity

2. The specific gravity of plastics shall

be expressed as the ratio of the weight of a given volume of the material at 25 C. (77 F.) to that of an equal volume of water at the same temperature, and shall be expressed thus:

Specific gravity, 25/25 C. (77/77 F.)...

## METHOD A.—FOR PLASTICS IN FINAL FORM FOR USE

### Apparatus

3. The apparatus shall consist of the following:

(a) *Analytical Balance.*—An analytical balance equipped with pan straddle or other stationary support.

(b) *Pycnometer.*—A pycnometer of one of the types shown in Fig. 1, for determining the specific gravity of kerosine when this is used as the displacement liquid in the determination.

(c) *Vacuum Desiccator.*—A glass desiccator, having a hole in the lid, and constructed to withstand a vacuum of one atmosphere.

(d) *Oil Vacuum Pump.*—An oil vacuum pump, with motor, to give a vacuum of 3 mm. or less.

(e) *Constant Temperature Bath.*—A

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

constant temperature bath, adjusted to  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.).

### Test Specimens

4. The test specimen for formed plastics shall consist of a piece cut from the material under test. The sides shall be made smooth by trimming, as in the case of thin sheet samples, or smoothing with abrasive cloth or paper for material of greater thickness, until the specimen is free from surface roughness which will entangle air bubbles when immersed in

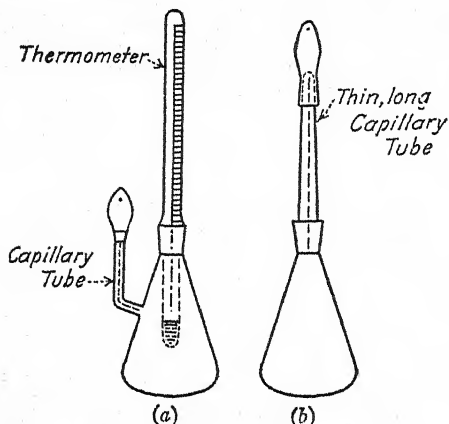


FIG. 1.—Two Types of Pycnometers.

liquid. The size of the specimen shall be such as to fit conveniently in the beaker used in the test, and may weigh from 1 to 5 g.

### Conditioning Test Specimens

5. Test specimens, whose change in specific gravity on conditioning may be greater than the accuracy required of the specific gravity determination, shall be conditioned before testing in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

### Procedure

6. (a) The balance shall first be tared

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

with a piece of fine wire, sufficiently long to reach from the hook on one of the pan supports to the pan straddle or other beaker support. The test specimen shall then be attached to the wire, so as to be suspended about 1 in. above the pan straddle or other beaker support, and weighed to the nearest 0.1 mg. This weight shall be called "a" (the weight in air). The specimen, still suspended by the wire, shall then be completely immersed in freshly boiled distilled water, or in kerosine, at a temperature of  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.), contained in a beaker of convenient size which is supported on the pan straddle, and any adhering air bubbles removed with a fine wire. The weight shall again be determined to the nearest 0.1 mg. This weight shall be called "b" (the weight in liquid).

(b) Where kerosine (or other nonaqueous liquid) is used as the immersing fluid, it is necessary to determine the specific gravity of the kerosine, as follows: Fill the pycnometer (Fig. 1) with freshly boiled distilled water and bring to a temperature of  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.). Remove air bubbles by placing in a vacuum desiccator and applying vacuum, then refilling (at  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.)) to make up for any change in volume. Weigh to the nearest 0.1 mg. Remove the water from the pycnometer, clean, dry, and weigh it empty. Then fill it with kerosine, bring again to 25 C. ( $77$  F.), remove the air bubbles as before, and replace any losses in volume (at  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.)) with kerosine. Reweigh to the nearest 0.1 mg. Calculate the specific gravity of the kerosine as follows:

$$\text{Sp. gr. of kerosine, } 25/25 \text{ C. (} 77/77 \text{ F.)} = \frac{\text{wt. of kerosine}}{\text{wt. of water}}$$

### Calculation

7. The specific gravity of the plastic shall be calculated from one of the two following formulas:

(a) *When water is the immersing medium:*

$$\text{Sp. gr., 25/25 C. (77/77 F.)} = \frac{a}{a - b}$$

where:

$a$  = weight in grams of sample in air, and  
 $b$  = weight in grams of sample in water at 25 C. (77 F.).

(b) *When kerosine (or other nonaqueous fluid) is the immersing medium:*

$$\text{Sp. gr., 25/25 C. (77/77 F.)} = \frac{a}{a - b}$$

× (sp. gr. of kerosine 25/25 C. (77/77 F.))

where:

$a$  = weight in grams of sample in air, and  
 $b$  = weight in grams of sample in kerosine (or other nonaqueous fluid) at 25 C. (77 F.).

METHOD B.—FOR PLASTICS IN THE FORM OF MOLDING POWDERS, FLAKE, OR THE LIKE

#### Apparatus

8. The apparatus shall be the same as described in Method A (see Section 3).

#### Test Specimens

9. Since molding powder or flake are rarely used under standard conditions, the specific gravity may be determined under any conditions desirable, that is, as received, as conditioned under standard conditioning procedures, as dried, etc., provided that the conditions be stated. If conditioning includes immersion in the liquid, the time and temperature shall be stated.

#### Procedure

10. (a) The pycnometer, previously standardized according to Section 6 (b), shall be weighed empty, a suitable quantity of the material (1 to 5 g.) added and the pycnometer reweighed. Boiled dis-

tilled water (or, for water soluble or sensitive materials, kerosine) shall then be added to cover the test specimen, and the pycnometer and contents placed in the vacuum desiccator. The lid of the vacuum desiccator shall be put in place and the vacuum pump connected and started. Vacuum shall be applied to the material in this manner to remove all air from the water (or kerosine) suspension of the test specimen. When all the air has been removed from the specimen, the vacuum shall be broken, the desiccator opened, and the pycnometer filled with the appropriate liquid, placed in the constant temperature bath, and allowed to remain until the pycnometer and contents have reached a temperature of  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.). The pycnometer shall then be filled exactly to the tip of the side arm by adding or removing the appropriate liquid, the outside of the pycnometer wiped dry, and the pycnometer and contents weighed.

(b) The pycnometer shall be emptied, cleaned, and filled with boiled distilled water, the air removed as in Paragraph (a), the temperature brought to  $25 \pm 0.1$  C. ( $77 \pm 0.2$  F.), and the pycnometer and contents reweighed. If kerosine was used as the fluid in the test, the process shall be repeated with the kerosine.

#### Calculations

11. The specific gravity of the material shall be calculated (Note) as follows:

$$\text{Sp. gr., 25/25 C. (77/77 F.)} = \frac{a \times d}{b - c + a}$$

where:

$a$  = weight in grams of specimen in pycnometer.

$b$  = weight in grams of pycnometer filled with displacement liquid.

$c$  = weight in grams of pycnometer containing specimen and filled with displacement liquid, and

$d$  = specific gravity of displacement liquid. If the liquid is kerosine, calculate as described in Section 7 (b); if water is used as the liquid the value of  $d$  will be unity.

NOTE.—The weight of the liquid having a volume equal to that of the test specimen is  $(b - c + a)$  and the volume of this weight of liquid is  $\frac{b - c + a}{d}$ .

#### Precautions

12. Before a new desiccator is used for the first time, it shall be wrapped in a towel, placed in a heavy wooden box, and tested under the vacuum to be used, the operator being adequately protected by the proper safety devices (goggles, safety shield, etc.). The same precautions shall be observed under operating conditions.

# *Tentative Method of Test for*

## SHORT-TIME STABILITY AT ELEVATED TEMPERATURES OF PLASTICS CONTAINING CHLORINE<sup>1</sup>



A.S.T.M. Designation: D 793 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers the procedure for determining the short-time stability at elevated temperatures of plastics containing chlorine.

### Apparatus

2. The apparatus, shown in Fig. 1, shall consist of the following:

(a) *Preheater Coil.*

(b) *Erlenmeyer Flask.*—An Erlenmeyer flask of 250 ml. capacity.

(c) *Glass Tube.*—A glass tube 6 mm. in diameter which is drawn to a point with an opening of 2 to 3 mm. and which extends to within  $\frac{1}{2}$  in. of the bottom of the flask.

(d) *Constant Temperature Bath.*—A constant temperature bath which can be controlled at  $180 \pm 2$  C.

(e) *Absorption Tube.*—A tube 18 to 20 mm. in diameter and 13 to 20 cm. in length for absorbing hydrogen chloride in the sodium hydroxide.

(f) *Nitrogen Cylinder.*

### Reagents

3. (a) *Sodium Hydroxide (0.1 N).*

(b) *Silver Nitrate (0.02 N).*

(c) *Ammonium Thiocyanate or Potassium Thiocyanate (0.02 N).*

(d) *Distilled Water.*

(e) *Ferric Nitrate.*—Approximately 5 per cent in distilled water.

### Test Specimens

4. The test specimen shall be 10 g. of the plastic cut into pieces so that one dimension is no larger than  $\frac{1}{16}$  in. The particle size of the specimen shall be agreed upon by the purchaser and the manufacturer.

### Procedure

5. (a) The 10-g. specimen, cut or shredded into small pieces, shall be spread evenly upon the bottom of the Erlenmeyer flask. The flask shall be placed in the constant temperature bath so that it is immersed in the heating medium to within 3 cm. of the top. The temperature shall be maintained at  $180 \pm 2$  C. ( $356 \pm 3.5$  F.) (Section 6 (b)). The flask shall be fitted with a rubber stopper

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.



holding two glass tubes, one being the glass inlet tube which shall extend nearly to the bottom of the flask. The inlet tube shall be connected to the preheater coil which in turn shall be connected to a nitrogen cylinder. The outlet tube shall be connected to the tube containing the absorbing solution. All glass tubing connections shall be butt joints held together by rubber tubing. The gas flow shall be adjusted so that the nitrogen bubbles through the absorbing tube at a rate of 2 to 4 bubbles per

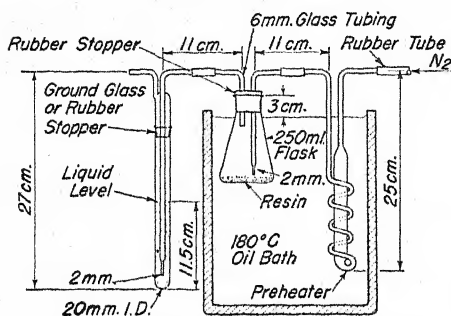


FIG. 1.—Assembly of Apparatus for Stability Test.

second. The gas shall be bubbled through the system for 30 min. and the evolved hydrogen chloride shall be absorbed in the tube containing 40 ml. of 0.1 *N* sodium hydroxide.

(b) At the end of the 30-min. period, the contents of the hydrogen chloride absorption tube and connecting tube shall be rinsed with distilled water into a beaker and acidified with nitric acid (sp. gr. 1.42). The chloride present shall be precipitated by adding 50 ml. of 0.02 *N* silver nitrate and the precipitated silver chloride shall be removed by filtration. The filtrate shall be tested with a drop of silver nitrate to be sure

all the chloride has been precipitated. The excess silver nitrate in the filtrate shall then be determined with 0.02 *N* ammonium thiocyanate (or potassium thiocyanate), using ferric nitrate as an indicator.

(c) *Blank*.—A blank determination shall be made on 40 ml. of the 0.1 *N* sodium hydroxide diluted with distilled water to a volume approximately equal to that used for the test on the specimen.

### Precautions

6. (a) The glass tubing and flask must be thoroughly dried before each determination.

(b) Control of the temperature within the limits specified is extremely important. If the temperature varies more than plus or minus 2 C. (3.5 F.) from 180 C. (356 F.), the determination shall be repeated.

(c) Since there is a possibility that the type of glass used might have an effect on the results obtained with some resins, the type of glass<sup>3</sup> used shall be stated in the report.

### Calculations

7. The short-time stability shall be calculated as follows:

$$M = [(A - B) - C] \times \text{normality of silver nitrate} \times 3.65$$

where:

*M* = short-time stability expressed as milligrams of hydrogen chloride evolved per gram of test specimen for a 10-g. specimen,

*A* = milliliters of silver nitrate,

*B* = milliliters of ammonium thiocyanate, and

*C* = milliliters of silver nitrate consumed by the blank.

<sup>3</sup> Pyrex glass has been found satisfactory.

# Tentative Method of Test for STIFFNESS IN FLEXURE OF NONRIGID PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 747 - 43 T

ISSUED, 1943.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. This method of test describes a procedure for determining the stiffness in flexure of nonrigid plastics. It is well suited for determining the relative flexibility over a wide range.

NOTE.—A discussion of the theory of the stiffness test will be found in the Appendix to this method.

## Apparatus

2. The apparatus for the stiffness test (Fig. 1) shall be of the cantilever beam bending type,<sup>3</sup> consisting essentially of the following:

(a) *Vise*.—A specimen vise,  $V$ , to which the pointer indicator,  $I_2$ , is attached, and which is capable of uniform clockwise rotation about the point  $O$  at a rate of 60 deg. of arc per min.

(b) *Weighing System*.—A pendulum weighing system, including an angular deflection scale, pointer indicator,  $I_1$ , bending plate,  $Q$ , for contacting the free

end of the specimen, and a series of detachable weights,  $M$ . This system shall be pivoted for nearly frictionless rotation about the point  $O$ .

(c) *Angular Deflection Scale*.—The angular deflection scale shall be calibrated in degrees of arc and shall indicate the angle through which the rotating vise has been turned relative to the pendulum system. This is the difference between the angle through which the vise has been turned and the angle through which the load pendulum has been deflected.

(d) *Load Scale*.—A fixed load scale which measures the deflection,  $\theta$ , of the pendulum system. It shall be calibrated to read directly  $100 L \sin \theta$ , where  $L$  is the distance between the center of rotation,  $O$ , and the center of the applied load,  $M$ . Thus,  $M$  times the load scale reading divided by 100 gives the bending moment directly.

(e) *Lubrication*.—For operation at subatmospheric temperatures, it is necessary to remove all the lubricant from the gear box, bearings, etc., of the apparatus. Kerosine may be used if found necessary.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by Committee E-10 on Standards, August 30, 1943.

<sup>3</sup> Olsen Stiffness Tester, Tour-Marshall and Olsen Design, described in U. S. Patent 2,049,235.

### Test Specimens

3. (a) Test specimens shall be strips cut from molded sheets of the material to be tested. They shall have a rectangular cross-section, and shall be cut with their longitudinal axis parallel to the direction of the calender grain, unless grain effects are specifically to be evaluated. The length, width, and thick-

ness shall be conditioned in accordance with the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618),<sup>4</sup> unless otherwise specified.

(b) Specimens to be tested at sub-atmospheric temperatures shall also be conditioned in air at the test temperature for 24 hr. prior to testing.

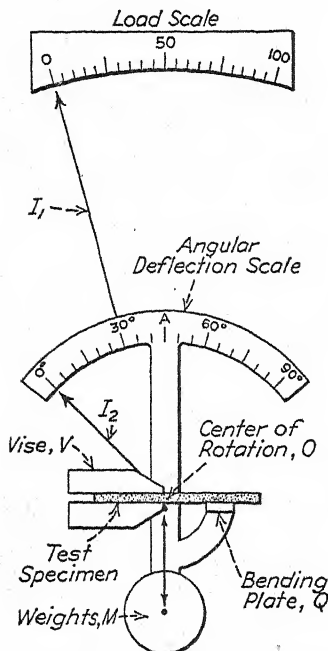


FIG. 1.—Mechanical System of Stiffness Tester.

ness of the specimen to be used will depend on the stiffness of the material and the capacity of the testing machine. Specimens shall have an even surface, but preferably not highly polished. If they exhibit a surface tackiness, they shall be dusted lightly with talc before being tested.

(b) Three specimens of the material shall be tested, and the results averaged.

### Conditioning Test Specimens

4. (a) All test specimens prior to

### Procedure

5. (a) The machine shall be set on an approximately level surface. The pendulum shall be weighted and, if necessary, the load scale adjusted to indicate zero. The bending pin or plate shall be set to the proper bending span after determining by trial the span required for the material to be tested. In general, the shortest span which will permit a complete test with the moment weights available shall be used. The motor shall be started and kept running throughout the test, since its vibration minimizes friction effects in the weighing system.

(b) The test specimen shall be clamped firmly in the vise with the center line approximately parallel to the face of the dial plate. By turning the hand crank, sufficient load shall be applied to the specimen to show a 1 per cent load reading and then the angle pointer shall be set to zero, and subsequent load readings reduced by one division.

(c) The motor engaging lever shall be held down and the load scale readings taken every 3 deg. up to 20 deg. and then every 10 deg. up to 90 deg. until the test is completed at 90 deg. deflection, or until fracture of the test specimen, or until the angle of bend or the load specified for the test is reached. Generally, data up to 30-deg. deflection are sufficient.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Calculations

6. (a) Plot the data on coordinate paper with the load scale reading as ordinate and the angular deflection as abscissa.

(b) The stiffness in flexure shall be calculated to three significant figures, as follows:

$$E = \frac{4S}{wd^3} \times \frac{M \times (\text{load scale reading})}{100\phi}$$

where:

$E$  = stiffness in flexure in pounds per square inch,

$S$  = span length in inches,

$w$  = specimen width in inches,

$d$  = specimen thickness in inches,

$M$  = calibrated weights applied to the pendulum system, and

$\phi$  = reading on angular deflection scale converted to radians.

NOTE.—The data when plotted, load scale reading *versus* angular deflection, is a curve generally with an initial straight line portion. If the straight line portion does not pass through the origin, it shall be translated parallel to itself until it does. The data consistent with some point on this straight line, which shall be extended, shall be used in the equation in Section 6(b).

### Report

7. The report shall include the following:

(1) Stiffness in flexure calculated in accordance with Section 6,

(2) All observed and recorded data on which the calculations are based,

(3) Complete identification of the material tested, including type, source, manufacturer's code number, form, surface, and dimensions, of the test specimens.

(4) Test room temperature, and

(5) Date of test.

## APPENDIX

### Theory of Operation of Stiffness Tester,<sup>3</sup> One-Half Inch Pound Capacity

A1. The mechanical system (Fig. 1) consists of several parts, as follows:

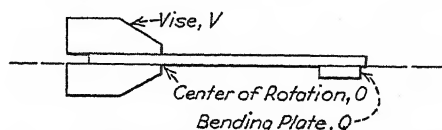
(a) The angular deflection scale,  $A$ , the pointer indicator,  $I_1$ , the bending plate,  $Q$ , and the mass,  $M$ , are rigidly attached to form a pendulum system pivoted for nearly frictionless rotation about the point  $O$ .

(b) The angular deflection scale,  $A$ , is calibrated in degrees of arc and indicates the angle through which the rotating vise has been turned relative to the point of load application on the test specimen. This is the difference between the angle through which the vise has been turned and the angle through which the load pendulum has been deflected.

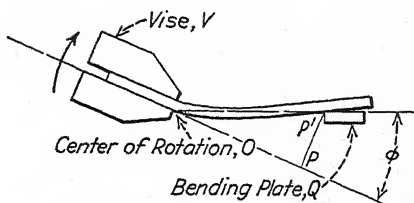
A2. The load scale which is fixed measures the deflection,  $\theta$ , of the pendulum system. It is calibrated to read directly  $100 L \sin \theta$ , where  $L$  (2 in.) is the distance between the center of rotation,  $O$ , and the center of the applied load,  $M$ . Thus,  $M$  multiplied by the load scale reading divided by 100 gives the bending moment.

A3. The specimen vise,  $V$ , to which the indicator,  $I_2$ , is attached, rotates in a clockwise direction about the point  $O$  to bend the specimen against the plate,  $Q$ .

A4. At the start of a test the specimen is mounted as shown in Fig. 2 (a). The indicator



(a) Starting Position



(b) Position During Test

FIG. 2.—Positions of Specimen.

$I_2$ , reads zero on the angular deflection scale,  $A$ , and the pointer indicator,  $I_1$ , indicates zero on the load scale. During test, the conditions

are as illustrated in Fig. 2 (b). The point  $P$  on the specimen has undergone a deflection which for small angles is given approximately by:

$$PP' = \frac{(ML \sin \theta)S^2}{3EI} \dots\dots\dots (1)$$

where:

$ML \sin \theta$  = moment of force which bends specimen,

$S$  = span length,

$E$  = modulus of elasticity in flexure, and

$I$  = moment of inertia of specimen cross-section which is calculated

as:  $\frac{wd^3}{12}$ , where  $w$  is the specimen width, and  $d$  the specimen thickness.

Dividing by  $S$ , the angle  $\phi$  (Fig. 2 (b)) which is registered on the angular deflection scale,  $A$ , is given by:

$$\phi = \frac{PP'}{S} = \frac{(ML \sin \theta)S}{3EI} \dots\dots\dots (2)$$

From Eq. (2) the modulus of elasticity (Note) or stiffness in flexure becomes:

$$E = \frac{(ML \sin \theta)S \times 100}{300I\phi} \dots\dots\dots (3)$$

or

$$E = \frac{4S}{wd^3} \times \frac{ML \sin \theta \times 100}{100\phi} \dots\dots\dots (4)$$

where:

$\phi$  = angular deflection on scale  $A$  converted to radians,

$100 L \sin \theta$  = load scale reading,

$ML \sin \theta$  = bending moment of specimen,

$S$  = specimen span,

$w$  = specimen width, and

$d$  = specimen thickness.

NOTE.—Under actual test conditions the deformation has both elastic and plastic components. The test method does not distinguish between or separate these, hence, a true elastic modulus is not calculable. Instead, an apparent value is obtained and is defined for purposes of this test as the stiffness of the material in flexure.

# *Tentative Method of Test for*

## TENSILE PROPERTIES OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 638 - 46 T

ISSUED, 1941; REVISED, 1942, 1944, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method is intended for use in determining the comparative tensile properties of organic plastics in the form of test specimens of standard shape and when tested under defined conditions of pretreatment, temperature, humidity, and testing machine speed. (Notes 1, 2, 3, and 4.)

### Definitions of Terms<sup>3</sup>

2. (a) *Tensile Stress* (nominal) is the tensile load per unit area of original cross-section, within the gage boundaries, carried by the test specimen at any time during a tension test. It is expressed in pounds per square inch.

(b) *Extension* is the change in length produced in a longitudinal section of a test specimen, measured between fixed

gage points on the specimen, by a tensile load. It is expressed in inches.

(c) *Strain* (or *Unit Extension*) is the ratio of the extension to the original length of the measured elongating section of the test specimen, that is, the change in length per unit of original length. It is expressed as a dimensionless ratio.

(d) *Tensile Strength* is the maximum tensile load per unit area of original cross-section, within the gage boundaries, carried by a test specimen of the type herein described during a tension test. It is expressed in pounds per square inch.

(e) *Percentage Elongation* is the extension recorded at the moment of rupture of the specimen expressed as a percentage of the original length of the measured elongating section. It is equal to one hundred times the strain measured at the moment of rupture of the specimen.

(f) *Mean Rate of Stressing* is, for report purposes, the tensile stress in pounds per square inch of original cross-section, carried by the test specimen at the moment when the strain is 0.02, or at the moment of rupture if this occurs at a strain below 0.02, divided by the

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> A more complete list of definitions of terms and symbols relating to the tension testing of plastics and related materials is being proposed to A.S.T.M. Committee E-1 on Methods of Testing and Committee E-8 on Nomenclature and Definitions, and is given in the Appendix. Attention is also directed to the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6), see p. 487.

time in seconds required for the attainment of this tensile stress and this strain, measured from the beginning of application of the load. It is expressed in pounds per square inch per second. (Note 5.)

(g) *Mean Rate of Straining* is, for report purposes, the rate of change of strain with time. It may best be determined by calculation from the slope of a strain-time curve, plotted for the purpose, using special data of strain recorded at definite intervals of time during the course of a test. It is expressed in dimensionless units per second.

(h) *Elastic Modulus* or *Young's Modulus* is the ratio within the elastic limit of stress to corresponding strain. (Note 6.) It is expressed in pounds per square inch.

(i) *Offset Yield Stress* is the stress at which the stress-strain curve departs from linearity by a specified percentage of deformation (offset). The method for determining the offset yield stress is prescribed in the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6)<sup>4</sup> under definition of yield strength, Paragraph 2 (a) on "Offset Method." The amount of offset shall always be stated, for example 0.2 per cent offset yield stress.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Testing Machine*.—A testing machine of the constant-rate-of-crosshead-movement type and comprising essentially the following:

(1) *Fixed Member*.—A fixed or essentially stationary member carrying one grip.

(2) *Movable Member*.—A movable member carrying a second grip.

(3) *Grips*.—Grips for holding the test specimen between the fixed member and the movable member. These must be

of the self-aligning type, that is, they must be attached to the fixed and movable member, respectively, in such a way that they will move freely into alignment as soon as any load is applied, so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly. The test specimen shall be held in such a way that slippage relative to the grips is prevented insofar as this is physically possible. For most thermoplastic materials, grips, the surfaces of which are deeply scored with a pattern similar to that of a coarse single-cut file, the serrations being about  $\frac{3}{32}$  in. apart and about  $\frac{1}{16}$  in. deep, have been found most satisfactory. Finer serrations have been found to be more satisfactory for harder, more brittle plastics, such as the thermosetting types.

(4) *Drive Mechanism*.—A drive mechanism for imparting to the movable member a uniform, controlled velocity with respect to the stationary member, this velocity to be regulated as specified in Section 7.

(5) *Load Indicator*.—A suitable load-indicating mechanism capable of showing the total tensile load carried by the test specimen when held by the grips. This mechanism shall be essentially free from inertia-lag at the specified rate of testing and shall indicate the load with an accuracy of plus or minus 1 per cent of the indicated value, or better. The accuracy of the testing machine shall be verified in accordance with the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials<sup>4</sup> (Note 7).

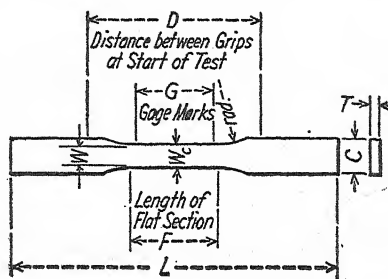
(6) The fixed member, movable member, drive mechanism, and grips shall be constructed of such materials and in such proportions that the total elastic

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



longitudinal strain of the system constituted by these parts does not exceed one per cent of the total longitudinal strain between the two gage marks on the test specimen at any time during

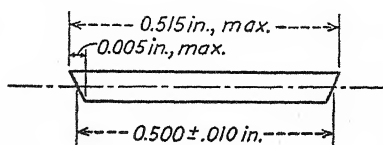
It is desirable, but not essential, that this instrument automatically record this distance (or any change in it) as a function of the load on the test specimen or of the elapsed time from the



Dimension, in.	Thickness, $T$ , in. <sup>d</sup>					Tolerances, in.
	$\frac{1}{4}$ in. or under		Over $\frac{1}{4}$ to $\frac{1}{2}$ in., incl.		Over $\frac{1}{2}$ to 1 in., incl.	
	Type I	Type II <sup>c</sup>	Type I	Type II <sup>c</sup>	Type I	
C—Width overall.....	0.750	0.750	1.125	1.125	1.500	$\pm 0.016$
W—Width of flat section <sup>a, b</sup> .....	0.500	0.250	0.750	0.375	1.000	$\pm 0.010$
F—Length of flat section.....	2.25	2.25	2.25	2.25	2.25	$\pm 0.016$
G—Gage length <sup>c</sup> .....	2.00	2.00	2.00	2.00	2.00	$\pm 0.016$
D—Distance between grips <sup>c</sup> .....	$4\frac{1}{2}$	$5\frac{1}{4}$	$4\frac{1}{2}$	$5\frac{1}{4}$	$5\frac{1}{4}$	$\pm \frac{1}{8}$
L—Length overall.....	$8\frac{1}{2}$	$9\frac{3}{8}$	$9\frac{3}{4}$	$10\frac{3}{8}$	12	minimum
Rad.—Radius of fillet.....	3	3	3	3	3	minimum

<sup>a</sup> The width at the center  $W_c$  shall be plus 0.000 in., minus 0.004 in. compared with width  $W$  at other parts of the reduced section. Any reduction in  $W$  at the center shall be gradual, equally on each side so that no abrupt changes in dimension result.

<sup>b</sup> For molded specimens, a draft of not over 0.005 in. may be allowed for the specimens 0.125 in. in thickness, and this should be taken into account when calculating width of the specimen. Thus, the cross-section of the molded specimen would be as follows:



<sup>c</sup> Test marks only.

<sup>d</sup> Thickness,  $T$ , shall be  $0.125 \pm 0.015$  in. for molded specimens and for other specimens where possible. If specimens are machined from sheets or plates, thickness,  $T$ , may be the thickness of the sheet or plate provided this does not exceed 1 in. For sheets of nominal thickness greater than 1 in. the specimens shall be machined to  $1.000 \pm 0.010$  in. in thickness. For sheets of nominal thickness between 1 and 2 in. approximately equal amounts shall be machined from each surface. For thicker sheets both surfaces of the specimen shall be machined and the location of the specimen with reference to the original thickness of the sheet, shall be noted. Tolerances on thickness less than 1 in. shall be those standard for the grade of material tested.

<sup>e</sup> Type I specimens shall be used whenever possible. Type II specimens shall be used only for materials with which the Type I specimen does not give satisfactory breaks in gage length, such as resin-impregnated compressed laminated wood.

FIG. 1.—Tension Test Specimens for Sheet, Plate, and Molded Plastics.

the test and at any load up to the rated capacity of the machine.

(b) *Extension Indicator*.—A suitable instrument for determining the distance between two fixed points on the test specimen at any time during the test.

start of the test, or both. This instrument shall be essentially free of inertia lag at the specified rate of loading, and shall be accurate to plus or minus one per cent of strain or better.

(c) *Micrometers*.—Suitable microm-

eters, reading to at least  $0.001 \pm 0.000$  in., for measuring the width and thickness of the test specimens.

(d) *Oven and Desiccators*.—A circulation air oven, adjustable to  $50 \pm 3$  C. ( $122 \pm 5$  F.), and desiccators containing anhydrous calcium chloride (or other suitable desiccant) are required for conditioning some materials.

### Test Specimen

4. (a) *Sheet, Plate, and Molded Plastics*.—The test specimen shall conform to the dimensions shown in Fig. 1. This specimen may be prepared by machining operations from materials in sheet, plate,

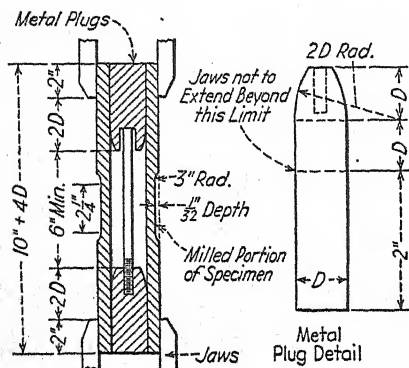


FIG. 2.—Tension Test Specimens for Tubes.

slab, or similar form, or it may be prepared by molding the material to be tested.

(b) *Tubes*.—The test specimen shall be as shown in Fig. 2. The length shall be 10 in. plus four times the inside diameter. When the wall thickness of the tube is  $\frac{1}{8}$  in. or greater, a groove  $2\frac{1}{4}$  in. in width in the flat portion and  $\frac{1}{32}$  in. in depth, with corners rounded to a radius of 3 in., shall be machined around the outside of the specimen at the center. Steel or brass plugs, having diameters such that they will fit snugly inside the tube and having a length of 2 in. plus twice the diameter, shall be placed in the ends of the specimen to prevent crushing.

They can be located in the tube conveniently by separating and supporting them on a metal rod, the lower end of which is screwed into the lower plug and the upper end of which rests loosely in a socket. Details of plugs and test assembly are shown in Fig. 2.

(c) *Rods*.—The test specimen as shown in Fig. 3 shall be about 12 in. in length. When the diameter of the rod is  $\frac{1}{4}$  in. or greater, a groove  $2\frac{1}{4}$  in. in width in the flat portion and  $\frac{1}{32}$  in. in depth, with corners rounded to a radius of 3 in., shall be machined around the specimen at the center.

(d) All surfaces of the specimen shall be free from visible flaws, scratches, or imperfections. Marks left by coarse

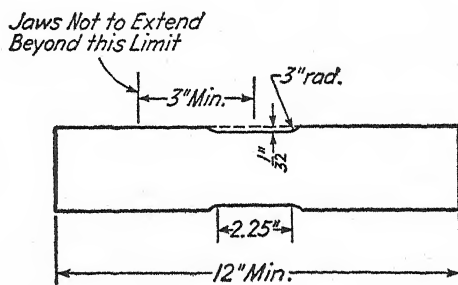


FIG. 3.—Tension Test Specimens for Rods.

machining operations shall be carefully removed with a fine file or abrasive and the filed surfaces shall then be smoothed with abrasive paper (No. 00 or finer). The finishing sanding strokes shall be made in a direction parallel to the long axis of the test specimen. All flash shall be removed from a molded specimen, taking great care not to disturb the molded surfaces. In machining a specimen, undercuts that would exceed the dimensional tolerances shown in Fig. 1 shall be scrupulously avoided. Care shall also be taken to avoid other common machining errors, such as those shown in Fig. 8 of the Standard Methods of Tension Testing of Metallic Materials (A.S.T.M. Designation: E 8 - 36) of the

American Society for Testing Materials.<sup>5</sup> (Note 8.)

(e) If it is necessary to place gage marks on the specimen, this shall be done with a soft, fine wax crayon or with India ink. Gage marks shall not be scratched into the surface of the specimen with a sharp instrument, because this causes weakness and premature fracture as a result of concentrations of stresses.

(f) When testing materials that may be suspected of anisotropy, duplicate sets of test specimens shall be prepared having their long axes respectively parallel with, and normal to the suspected direction of anisotropy. (Note 8.)

#### Conditioning

5. (a) Plastic materials may be preconditioned for testing under either "dry" or "standard" atmospheric conditions. (Note 9.) In general, the preconditioning method is chosen on the basis of the rapidity with which the tensile properties of the material in question vary with changes in atmospheric humidity and temperature. Thus, it is known that the tensile properties of cellulose derivatives and of some polyvinyl acetal resins change with comparative rapidity when subjected to rapidly changing temperature or humidity, whereas the tensile properties of polystyrene, polymethacrylic acid esters, phenol-formaldehyde, and urea-formaldehyde plastics are only slowly affected by such atmospheric changes. Hence, the latter group of materials has most frequently been prepared for testing by "dry" preconditioning methods, whereas the former group has most frequently been prepared by "standard" preconditioning methods.

(b) "Standard" Conditioning Procedure.—Specimens shall be preconditioned for at least 14 days in an enclosure maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and

$50 \pm 2$  per cent relative humidity prior to testing. The actual tests shall be conducted at the same atmospheric conditions. (Note 10.)

(c) "Dry" Conditioning Procedure.—Specimens shall be preconditioned for a period of 48 hr. in a circulating air oven at a temperature of  $50 \pm 3$  C. ( $122 \pm 5$  F.). They shall then be removed from the oven, cooled in a desiccator over anhydrous calcium chloride (or an equivalent desiccant) for a period of at least 16 hr. prior to testing. The test shall be conducted in an enclosure maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity. A definite time interval, previously decided upon by cooperating laboratories, shall be allowed to elapse between removal of the specimens from the desiccator and the application of load in the test.

(d) Special conditioning procedures may be used by agreement between cooperating laboratories.

(e) If no conditioning method is specified, the "standard" conditioning procedure shall be used (Note 11).

#### Number of Test Specimens

6. (a) At least five specimens shall be tested for each sample in the case of isotropic materials.

(b) Ten specimens, five normal to, and five parallel with the principal axis of anisotropy, shall be tested for each sample in case of anisotropic materials.

(c) Specimens that break at some obvious fortuitous flaw or that do not break between the predetermined gage marks shall be discarded and retests made, unless such flaws constitute a variable the effect of which it is desired to study.

(d) Results that deviate from the mean value of all tests should be rejected if the deviation of the doubtful value is more than five times the average deviation from the mean obtained by excluding the doubtful value. Such

<sup>5</sup>1939 Book of A.S.T.M. Standards, Part I, p. 750.

doubtful values shall be discarded and retests made, unless the degree of variability is a factor that is being studied.

### Speed of Testing

7. (a) Speed of testing is the velocity of separation of the two members (or grips) of the testing machine when running idle (under no load).

(b) The standard speed of testing shall be 0.20 to 0.25 in. per min.

(c) If load-deformation data are being taken, as for determination of elastic modulus or for study of the initial portion of the stress-strain curve, the speed of testing shall be 0.05 in. per min. until the "yield point" is reached. The extension recorder shall then be removed, the rate of crosshead movement increased to the standard speed of 0.20 to 0.25 in. per min., and maintained at that speed until failure of the specimen. (Notes 12 and 13.)

### Procedure

8. (a) The actual testing shall be carried out in an atmosphere maintained at  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity.

(b) The width and thickness of the flat specimen (Fig. 1) shall be measured to the nearest 0.001 in. at several points along its length. The diameter of rod specimens, and the inside and outside diameters of tube specimens, shall be measured to the nearest 0.001 in. at a minimum of two points 90 deg. apart; these measurements shall be made at the groove for specimens so constructed. Record the minimum values of cross-sectional area so determined. Plugs shall be used in testing tube specimens as shown in Fig. 2.

(c) The specimen shall be placed in the grips of the testing machine, taking care to align the long axis of the specimen and the grips with an imaginary line joining the points of attachment of the grips to the machine. There shall be allowed 0.25 in. between the ends of

the gripping surfaces and the shoulders of the fillet of the flat test specimen; thus, the ends of the gripping surfaces should be 4.5 in. apart, as shown in Fig. 1, at the start of the test. The location for the grips on tube and rod specimens shall be as shown in Figs. 2 and 3. The grips shall be tightened evenly and firmly to the degree necessary to prevent slippage of the specimen during the test but not to the point where the specimen would be crushed.

(d) If stress-strain data are desired the procedure shall be as follows:

(1) Attach the extension recorder.

(2) Set the speed control at 0.05 in. per min. and start the machine.

(3) Record loads and corresponding deformations at appropriate even intervals of strain.

(4) Record the load carried by the specimen when the strain reaches 0.02 and the elapsed time from the start of the test until this point is reached. If rupture occurs before the strain reaches 0.02, record the elapsed time from the start of the test until the specimen breaks.

(5) After the "yield point" has been reached (or after the strain has passed 0.02), increase the speed to 0.20 to 0.25 in. per min. and allow the machine to run at this speed until the specimen breaks.

(6) Record the maximum load carried by the specimen during the test (usually this will be the load at the moment of rupture).

(7) Record the extension at the moment of rupture of the specimen.

(e) If only tensile strength and elongation data are desired, the procedure shall be as follows:

(1) Set the speed control at 0.20 to 0.25 in. per min. and start the machine.

(2) Record the load carried by the specimen when the strain reaches 0.02 and the elapsed time from the start of the test until this point is reached. If

rupture occurs before the strain reaches 0.02, record the elapsed time from the start of the test until the specimen breaks.

(3) Record the maximum load carried by the specimen during the test (usually this will be the load at the moment of rupture).

(4) Record the extension at the moment of rupture of the specimen.

### Calculations

9. (a) *Tensile Strength* shall be calculated by dividing the maximum (or breaking) load in pounds by the original minimum cross-sectional area of the specimen in square inches. The result shall be expressed in pounds per square inch and reported to three significant figures.

(b) *Percentage Elongation* shall be calculated by dividing the extension at the moment of rupture of the specimen by the original distance between gage marks and multiplying by one hundred. The percentage elongation shall be reported to two significant figures.

(c) *Mean Rate of Stressing* shall be calculated by dividing the tensile load carried by the specimen when the strain reaches 0.02 or at the moment of rupture, whichever occurs first, by the original minimum cross-sectional area of the specimen, and then dividing this result by the time in seconds, measured from the beginning of the test, required to attain this tensile load and strain. The result shall be expressed in pounds per square inch per second and reported to three significant figures.

(d) *Mean Rate of Straining* shall be calculated from a strain-time curve, plotted for the purpose, by selecting any convenient point on the curve and dividing the strain represented by the point by the corresponding time. The result shall be expressed as a dimensionless ratio per second (units per second) and reported to three significant figures.

(e) *Elastic Modulus* shall be calcu-

lated by drawing a tangent to the initial linear portion of the stress-strain curve, selecting any point on this straight line and dividing the tensile stress (nominal) represented by the point by the corresponding strain. The result shall be expressed in pounds per square inch and reported to three significant figures. (Note 14.)

(f) For each series of tests, the arithmetic mean of all values obtained shall be calculated to three significant figures and reported as the "average value" for the particular property in question.

(g) The deviation of each value from the "average value" shall be calculated and the arithmetic mean of these deviations determined. This arithmetic mean shall be reported to two significant figures as the "average deviation" of the particular series of results.

### Report

10. The report shall include the following:

(1) Complete identification of the material tested, including type, source, manufacturer's code numbers, form, principal dimensions, previous history, etc.,

(2) Method of preparing test specimens,

(3) Type of test specimen and dimensions,

(4) Conditioning procedure used,

(5) Atmospheric conditions in test room,

(6) Number of specimens tested,

(7) Speed of testing,

(8) Mean rate of stressing as calculated in Section 9 (c),

(9) Mean rate of straining as calculated in Section 9 (d),

(10) Tensile strength, average value and average deviation,

(11) Percentage elongation, average value and average deviation,

(12) Elastic modulus, average value and average deviation, and

(13) Date of test.

## EXPLANATORY NOTES

NOTE 1.—This method is not well adapted to the testing of plastics in the form of thin sheets or films; a method for the testing of such forms is given in the Tentative Method of Test for Tensile Properties of Thin Plastic Sheets and Films (A.S.T.M. Designation: D 882).<sup>4</sup> Neither is this method particularly suitable for the testing of rubber or rubber-like polymers (elastomers), although the position of such materials as organic plastics is recognized.

NOTE 2.—This method may be used for testing phenolic resin molded or laminated materials where comparative results are desired. However, where correlation with previous data is an object, such materials should be tested in accordance with the following methods of the American Society for Testing Materials:

Tentative Methods of Testing Molded Materials Used for Electrical Insulation (A.S.T.M. Designation: D 48),<sup>4</sup>

Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229),<sup>4</sup> and

Tentative Method of Test for Tensile Strength of Molded Electrical Insulating Materials (A.S.T.M. Designation: D 651).<sup>4</sup>

NOTE 3.—It is realized that a material can not be tested without also testing the method of preparation of that material. Hence, when comparative tests of materials *per se* are desired, the greatest care must be exercised to insure that all samples are prepared in exactly the same way. Similarly, for referee or comparative tests of any given series of specimens, care must be taken to secure the maximum degree of uniformity in details of preparation, treatment, and handling.

NOTE 4.—This method is not intended to cover precise physical procedures. It is recognized that the constant-rate-of-crosshead-movement type of test leaves much to be desired from a theoretical standpoint, that wide differences may exist between rate of crosshead movement and rate of strain between gage marks on the specimen, and that the testing speeds specified disguise important effects characteristic of materials in the plastic state. Further, it is realized that variations in the thicknesses of test specimens, which are permitted by these procedures, produce variations in the surface-volume ratios of such specimens, and that these variations may influence the test results. Hence, where directly comparable results are desired, all samples should be of equal thickness. Special additional tests should be used where more precise physical data are needed.

NOTE 5.—It is realized that "mean rate of stressing" as defined in these methods has only limited physical significance. It does, however, roughly describe the average rate at which most of the stress carried by the test specimen is applied and for that portion of the stress-strain curve in which principal stressing occurs. It is affected by the elasticity of the materials being tested but is fairly accurately determined by the method described. It can, if desired, be determined more precisely by calculation from load-time data, recorded especially for the purpose during a test.

NOTE 6.—Since the existence of a true elastic limit in plastics, as in many other organic materials and in many metals, is debatable, the propriety of applying the term "elastic modulus" in its quoted generally accepted definition to describing the "stiffness" or "rigidity" of a plastic has been seriously questioned. The exact stress-strain characteristics of plastic materials are highly dependent on such factors as rate of application of stress, temperature, previous history of specimen, etc. However, stress-strain curves for plastics determined as described in this method almost always show a linear region at low stresses, and a straight line drawn tangent to this portion of the curve permits calculation of an elastic modulus of the usually defined type. Such a constant is useful if its arbitrary nature and dependence on time, temperature, and similar factors is realized.

NOTE 7.—Experience has shown that many testing machines now in use are incapable of maintaining accuracy for as long as the periods between inspection recommended in Standard Methods E 4.<sup>4</sup> Hence, it is recommended that each machine be studied individually and verified as often as may be found necessary. It will frequently be necessary to perform this function daily.

NOTE 8.—Before testing, all transparent specimens should be given a polariscopic inspection and those which show atypical or concentrated strain patterns should be rejected unless these "initial" strains constitute a variable the effect of which it is desired to study.

NOTE 9.—In general, the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618) of the American Society for Testing Materials<sup>4</sup> will be found useful.

NOTE 10.—In cases of extreme urgency, where the 14-day conditioning period is intolerable, the conditioning may, by agreement between co-operating laboratories, be reduced to a minimum of 48 hr.

NOTE 11.—It is obvious that when exact comparative tests are desired, comparable conditioning procedures should be employed. It is urged that "standard" conditions be employed for all tests intended for data, specification, or similar purposes, in so far as this is practicable.

NOTE 12.—Such composite tests should not

laminated thermosetting materials, may be tested at a speed of 0.05 in. per min., but this should be stated in the report.

NOTE 14.—Since stress-strain data are relatively inaccurate at low values of stress and strain (near the start of the test), the truly linear portion of the curve usually will not pass through

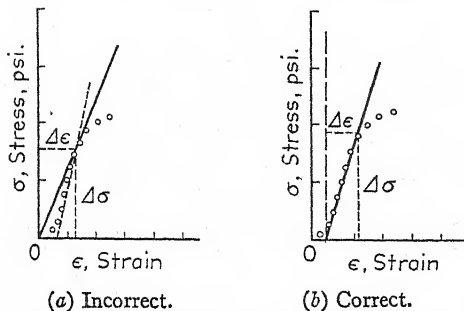


FIG. 4.—Measuring Strain from Stress-Strain Diagram.

be made when exact data on tensile strength and elongation are desired. Such tests should be made at the standard speed of 0.20 to 0.25 in. per min. from beginning to the end of the test.

NOTE 13.—Where correlation with previous data or with other methods, for example, the Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (A.S.T.M. Designation: D 229) of the American Society for Testing Materials,<sup>4</sup> is desired, materials of low ductility, such as molded or

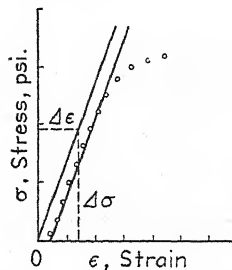


FIG. 5.—Modulus Calculations from Stress-Strain Diagram.

the origin. Care must be exercised to draw a straight line which truly represents the slope of the curve below the elastic limit and to measure strain values from the point at which this line intersects the zero stress axis for modulus calculations (see Fig. 4). Alternatively, a true modulus line may be drawn through the linear portion of the curve, a second line, parallel to this line, may then be constructed through the origin, and values of stress and strain for modulus calculations may be obtained therefrom (see Fig. 5).

(See Appendix, p. 908)



## APPENDIX

## DEFINITIONS OF TERMS AND SYMBOLS RELATING TO TENSION TESTING OF PLASTICS

## DEFINITIONS

1. *Tensile Strength* is the maximum tensile load per unit area of original cross-section, within the gage boundaries, required to break a test specimen. ("Tensile load" is interpreted to mean the maximum tensile load sustained by the specimen during the test, whether or not this coincides with the tensile load at the moment of rupture.) It is expressed in pounds per square inch.
2. *True Ultimate (or Tensile) Strength* is the tensile load per unit of minimum cross-sectional area within the gage boundaries, measured at the moment of rupture, required to break a test specimen. It is expressed in pounds per square inch.
3. *Tensile Stress* (nominal) is the tensile load per unit area of original cross-section, within the gage boundaries, carried by the test specimen at any given moment. It is expressed in pounds per square inch.
4. *True Tensile Stress* is the tensile load per unit of minimum cross-sectional area within the gage boundaries carried by the test specimen at any given moment. It is expressed in pounds per square inch.
5. *Extension* is the change in length produced in a longitudinal section of a test specimen, measured between fixed gage points on the specimen, by a tensile load. It is expressed in inches.
6. *Strain* is the ratio of the extension to the original length of the measured elongating section of the test specimen, that is, the change in length per unit original length. It is expressed as a dimensionless ratio. (Also known as *Longitudinal Tensile Strain*.)
7. *Unit Extension* is synonymous with *Strain*.
8. *Ultimate Extension* is the extension recorded at the moment of rupture of the test specimen.
9. *Ultimate Strain* is the unit extension recorded at the moment of rupture of the test specimen.
10. *Ultimate Unit Extension* is synonymous with *Ultimate Strain*.
11. *Percentage Elongation* is the extension recorded at the moment of rupture of the specimen expressed as a percentage of the original length of the measured elongating section. It is equal to one hundred times the strain measured at the moment of rupture of the specimen.
12. *Percentage Ultimate Extension* is synonymous with *Percentage Elongation*.
13. *Nominal Reduction of Area* is the difference between the original cross-sectional area of the test specimen and the cross-sectional area measured at the point of rupture after breaking the specimen in tension. It is expressed in square inches.
14. *Nominal Unit Reduction of Area* is the ratio of the nominal reduction in area to the original cross-sectional area of the test specimen, that is, the nominal reduction in area per unit original cross-sectional area. It is expressed as a dimensionless ratio.
15. *Nominal Percentage Reduction of Area* is the nominal reduction in area expressed as a percentage of the original cross-sectional area of the specimen. It is equal to the *Nominal Unit Reduction in Area* multiplied by one hundred.
16. *True Reduction of Area* is the difference between the original cross-sectional area of the test specimen and the minimum cross-section area within the gage boundaries at the moment of rupture in tension. It is expressed in square inches.
17. *True Unit Reduction of Area* is the ratio of the true reduction in area to the original cross-sectional area of the test specimen; that is, the true reduction in area per unit original cross-sectional area. It is expressed as a dimensionless ratio.
18. *True Percentage Reduction of Area* is the true reduction in area expressed as a percentage of the original cross-sectional area of the specimen. It is equal to the *True Unit Reduction in Area* multiplied by one hundred.
19. *Tensile Stress-Strain Curve* is the curve obtained by plotting *Tensile Stresses* as ordinates against corresponding *Longitudinal Tensile Strains (Unit Extensions)* as abscissas for the entire course of a tension test.
20. *True Tensile Stress-Strain Curve* is the curve obtained by plotting *True Tensile Stresses* as ordinates against corresponding *Longitudinal Tensile Strains (Unit Extensions)* as abscissae for the entire course of a tension test.
21. *Upper Yield Stress* is the first stress asymptote on the stress-strain curve (whether nominal or true stress should be stated) at which the slope of the curve becomes zero, that is, where the tangent to the curve becomes horizontal, when the test is conducted on a machine having a constant rate of crosshead movement.
22. *Lower Yield Stress* is the second stress asymptote on the stress-strain curve (whether nominal stress or true stress should be stated) at which the slope of the curve becomes zero,

that is, where the tangent to the curve becomes horizontal, when the test is conducted on a machine having a constant rate of crosshead movement.

NOTE.—At the *Upper Yield Stress* the stress-strain curve is concave downward; at the *Lower Yield Stress* the stress-strain curve is concave upward.

23. *Offset Yield Stress* is the stress at which the stress-strain curve departs from linearity by a specified percentage of deformation (offset). The method for determining the offset yield stress is prescribed in the Standard Definitions of Terms Relating to Methods of Testing (A.S.T.M. Designation: E 6)<sup>4</sup> under definition of yield strength, Paragraph 2 (a) on "Offset Method." The amount of offset shall always be stated, for example, 0.2 per cent offset yield stress.

24. *Rate of Loading* is the variation in tensile load carried by the specimen per unit time. It is expressed in pounds per second.

25. *Rate of Stressing* is the variation in tensile stress per unit time. It is expressed in pounds per square inch per second.

26. *True Rate of Stressing* is the variation in true tensile stress per unit time. It is expressed in pounds per square inch per second.

NOTE.—At the nominal plastic yield stress the instantaneous nominal rate of stressing becomes zero, but the instantaneous true rate of stressing may continue to have a positive value if the cross-sectional area is decreasing.

27. *Mean Rate of Stressing* is defined, for report purposes, as the *Tensile Stress* at which the *Strain* is 0.02, or at the moment of rupture if this occurs at a strain below 0.02, divided by the time required for the attainment of this stress, measured from the beginning of load application. It is expressed in pounds per square inch per second.

28. *Mean True Rate of Stressing* is defined, for report purposes, as the *True Tensile Stress* at which the *Strain* is 0.02, or at the moment of rupture if this occurs at a strain below 0.02, divided by the time required for the attainment of this true stress, measured from the beginning of load application. It is expressed in pounds per square inch per second.

29. *Rate of Straining* is the variation in longitudinal tensile strain per unit of time.

NOTE.—It is not synonymous with rate of crosshead movement.

30. *Elastic Limit* is the greatest tensile stress which a material is capable of carrying without a permanent deformation remaining upon complete release of the stress.

31. *Elastic Modulus* is the ratio, within the elastic limit of a material, of stress to corresponding strain.

## SYMBOLS

32. The following symbols may be used for the above terms:

- $W$  = Weight load,
- $\Delta W$  = Increment of weight load,
- $L$  = Distance between gage marks at any time,
- $L_o$  = Original distance between gage marks,
- $L_u$  = Distance between gage marks at moment of rupture,
- $\Delta L$  = Increment of distance between gage marks = extension,
- $A$  = Minimum cross-sectional area at any time,
- $A_o$  = Original cross-sectional area,
- $\Delta A$  = Increment of cross-sectional area,
- $A_u$  = Cross-sectional area at point of rupture, measured after breaking specimen,
- $A_T$  = Cross-sectional area at point of rupture, measured at the moment of rupture,
- $t$  = Time,
- $\Delta t$  = Increment of time,
- $\sigma$  = Tensile stress,
- $\sigma_T$  = True tensile stress,
- $\sigma_U$  = Ultimate (or tensile) strength,
- $\sigma_{UT}$  = True ultimate (or tensile) strength,
- $\epsilon$  = Strain (= unit extension),
- $\epsilon_U$  = Ultimate strain (= ultimate unit extension),

$\%El$  = Percentage elongation = percentage ultimate extension,

P.Y.S. ( $u$ ) = Upper yield stress (nominal),

P.Y.S. ( $L$ ) = Lower yield stress (nominal), and

$E$  = Elastic modulus.

33. Relations between these various terms may be defined as follows:

$$\sigma = \frac{W}{A_o}$$

$$\sigma_T = \frac{W}{A}$$

$$\sigma_U = \frac{W}{A_o} \quad (\text{where } W \text{ is breaking load})$$

$$\sigma_{UT} = \frac{W}{A} \quad (\text{where } W \text{ is breaking load})$$

$$\epsilon = \frac{\Delta L}{L_o} = \frac{L - L_o}{L_o}$$

$$\epsilon_U = \frac{L_u - L_o}{L_o}$$

$$\%El = \frac{L_u - L_o}{L_o} \times 100 = \epsilon_U \times 100$$

$$\text{Nominal reduction of area} = A_o - A_u$$

$$\text{Nominal unit reduction of area} = \frac{A_o - A_u}{A_o}$$

$$\text{Nominal percentage reduction of area} = \frac{A_o - A_u}{A_o} \times 100$$

$$\text{True reduction of area} = A_o - A_T$$

$$\text{True unit reduction of area} = \frac{A_o - A_T}{A_o}$$

$$\text{True percentage reduction of area} = \frac{A_o - A_T}{A_o} \times 100$$

$$\text{Rate of loading} = \frac{\Delta W}{\Delta t}$$

$$\text{Rate of stressing} = \frac{\Delta \sigma}{\Delta t} = \frac{\frac{\Delta W}{A_o}}{\Delta t}$$

$$\text{True rate of stressing} = \frac{\Delta \sigma_T}{\Delta t} = \frac{\frac{\Delta W}{A}}{\Delta t}$$

$$\text{Rate of straining} = \frac{\Delta \epsilon}{\Delta t} = \frac{\frac{\Delta L}{L_o}}{\Delta t}$$

For the special case where the volume of the test specimen does not change during the test, the following relations hold:

$$\sigma_T = \sigma(1 + \epsilon) = \sigma \left( \frac{L}{L_o} \right)$$

$$\sigma_{UT} = \sigma_U(1 + \epsilon_U) = \sigma_U \left( \frac{L_u}{L_o} \right)$$

$$A = \frac{A_o}{(1 + \epsilon)}$$

# *Tentative Method of Test for*

## TENSILE PROPERTIES OF THIN PLASTIC SHEETS AND FILMS<sup>1</sup>



A.S.T.M. Designation: D 882 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers a test for use in determining the tensile properties of organic plastics in the form of relatively thin sheets or films (less than 0.020 in. in thickness), when tested under defined conditions of pretreatment, temperature, humidity, and rate of stressing by a constant-rate-of-load method. It is not intended for use in testing rubber-like materials whose percentage elongation is greater than approximately 350 (Note).

NOTE.—Such materials may be tested in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412).<sup>3</sup>

### Definitions of Terms

2. (a) *Tensile Stress* (nominal) is the tensile load per unit area of original cross-section, within the gage boundaries, carried by the test specimen at any time during a tensile test. It is expressed in pounds per square inch.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) *Extension* is the change in length produced in longitudinal section of a test specimen, measured between fixed gage points on the specimen, by a tensile load. It is expressed in inches.

(c) *Strain* (or *Unit Extension*) is the ratio of the extension to the original length of the measured elongating section of the test specimen, that is, the change in length per unit of original length. It is expressed as a dimensionless ratio.

(d) *Tensile Strength* is the maximum tensile load per unit area of original cross-section, within the gage boundaries, carried by a test specimen of the type herein described during a tension test. It is expressed in pounds per square inch.

(e) *Percentage Elongation* is the extension recorded at the moment of rupture of the specimen expressed as a percentage of the original length of the measured elongating section. It is equal to one hundred times the strain measured at the moment of rupture of the specimen.

(f) *Rate of Stressing* is defined, for report purposes, as the rate of loading (Note) divided by the original cross-section of the specimen. It is expressed in pounds per square inch per minute.

NOTE.—The rate of loading may be determined for the constant rate of loading type machine (as required for this test) by timing a test with a stop watch and dividing the final load, as recorded by the chart, by the time required for the application of this load. Uniformity of loading rate, throughout the range of the machine, should be verified by actual observation.

(g) *Elastic Modulus* is the ratio within the proportional limit of stress to corresponding strain. It is expressed in pounds per square inch.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Testing Machine*.—Any suitable film testing machine of the constant rate of loading type. It should be equipped with a device for recording the tensile load carried by the specimen and recording the rate of jaw separation during the test. Both the stressing and recording mechanism should be essentially free from inertia lag at the specified rate of stressing, and frictional drag should be reduced to a minimum. The machine should indicate the load with an accuracy of plus or minus 2 per cent, or better. A device for varying the loading rate between approximately 50 and 300 lb. per min. should be included (Note 1).

NOTE 1.—A machine of the tilting-table type has been found satisfactory.

(b) *Grips*.—Flat, smooth steel grips should be used where possible and tests should be started with the grips separated a distance of 2 in. (Note 2).

NOTE 2.—Other types of gripping surfaces may be required for some types of plastics. For example, file-faced grips or grips lined with abrasive paper have been found necessary for some materials. Also it may be necessary in some cases to use an initial grip spacing of less than 2 in. Neither of these variations should be used unless it is impossible to obtain satisfactory results with the conditions recommended.

(c) *Micrometers*.—Suitable micrometers, or other thickness gages, reading to 0.0001 in. or less, for measuring the width and thickness of the test specimens.

(d) *Paper Cutter*.—A suitable paper cutter or trimmer for trimming the specimens to the proper width. It is important that the cutting edges of the cutter be kept sharp and free from scratches or nicks.

(e) *Conditioning Apparatus*.—Apparatus for maintaining suitable atmospheric conditions, as defined in the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup> Unless otherwise specified, the standard laboratory atmospheric condition of  $25 \pm 1$  C. ( $77 \pm 2$  F.) and  $50 \pm 2$  per cent relative humidity is required.

### Test Specimen

4. (a) The test specimen shall consist of a strip of uniform width and thickness, and 5 in. or more in length.

(b) The width of the specimen shall be not less than 0.25 in. but not greater than 1.0 in. and shall be chosen so as to allow failure to occur well within the load capacity of the machine. A combination of cross-section and loading rate which gives the proper rate of stressing shall be selected.

(c) A width-thickness ratio of greater than eight shall be used, if possible, since narrow strips emphasize the effect of edge strains or flaws incurred during cutting.

(d) The utmost care shall be exercised in cutting strip specimens to prevent nicks and tears in the edges which might give rise to premature fractures. The sheet shall be held firmly, and close to the knife edge. The blade shall be drawn through the sheet slowly but smoothly. The edges shall be parallel to within 2 per cent of the width.

(e) Gage marks shall be placed on the

specimen in order to obtain reliable elongation data (Note). This shall be done with a soft, fine wax crayon, or with india ink. Gage marks shall not be scratched onto the surface with a sharp instrument, because this causes weakness and premature fracture as a result of stress concentration.

NOTE.—The use of gage marks is necessary because materials with appreciable elongation pull away from the grips. As the specimen elongates, the accompanying reduction in area results in a loosening of material just inside the grips. This moves back into the grips as further elongation and reduction in area take place.

(f) In testing materials which may be suspected of anisotropy, duplicate sets of test specimens shall be prepared having their long axis respectively parallel with and normal to the suspected direction of anisotropy.

### Conditioning

5. Prior to testing, the specimens shall be conditioned in accordance with A.S.T.M. Methods D 618.

### Number of Test Specimens

6. (a) At least ten specimens shall be tested for each sample in the case of isotropic materials.

(b) Twenty specimens, ten normal to and ten parallel with the principal axis of anisotropy, shall be tested for each sample in case of anisotropic materials.

(c) Results on specimens which break at some obvious chance flaw or which break in the grips shall be discarded and retests made, unless such failures constitute a variable whose effect is being studied.

(d) Results on specimens which deviate markedly from the mean value of all tests shall be rejected if the deviation of the doubtful value is more than five times the average deviation from the mean value obtained by excluding the doubtful

result. Such doubtful results shall be discarded and retests made unless the degree of variability is a factor which is being studied (Note).

NOTE.—For some materials whose properties vary considerably throughout the sheet, as many as 50 specimen width from portions of the sheet shall be tested, if a reliable picture of properties is desired.

### Speed of Testing

7. The rate of stressing (Section 2 (f)), shall be held between the limits of 15,000 and 65,000 psi. per min. in all tests. After an appropriate specimen width has been selected (Section 4 (b)), the desired stressing rate shall be obtained by varying the loading rate until the ratio of loading rate to specimen cross-section is approximately 40,000 psi. per min.

### Procedure

8. (a) A specimen width shall be selected which will produce failure well within the load limit of the testing machine. If necessary, a few trial runs shall be made in order to make a proper selection.

(b) The loading rate shall be set at a value such that it equals the product of the standard stress rate (15,000 to 65,000 psi. per min.) and the cross-section (in square inches) of the specimen (Note 1).

NOTE 1.—Recommended widths and loading rates are shown in the following table:

Nominal Thickness of Film, in.	Recommended Width of Specimen, in.	Recommended Rate of Loading, lb. per min.	Recommended Rate of Stressing, psi. per min.
0.003 .....	0.5	60	40 000
0.005 .....	0.5	100	40 000
0.0075 .....	0.5	150	40 000
0.010 .....	0.5	200	40 000
0.0125 .....	0.5	250	40 000
0.015 .....	0.5	300	40 000
0.020 .....	0.375	300	40 000

(c) The thickness and width of the specimen shall be measured, reading the thickness to the nearest 0.0001 in. or

better, and the width to 0.001 in. or better, at several points along its length. The minimum cross-section found shall be recorded.

(d) The test specimen shall be placed in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine (Note 2). At least 1 in. of specimen shall be included in each grip. The grips shall be tightened evenly and firmly to the degree necessary to prevent unnecessary slipping of the specimen during the test; adjustment with a torque wrench to 50 in.-lb. is recommended.

NOTE 2.—Great care in alignment is necessary to prevent failure by tearing rather than by true tension, and tests should be watched closely to make sure that tearing failures do not occur.

(e) A light line shall be ruled with ink or crayon, across the specimen adjacent to the edge of either grip.

(f) The recording pen shall be adjusted so that it starts at the proper point on the load-extension chart.

(g) While the specimen is being stressed, the displacement of the line described in Paragraph (e) shall be followed with respect to the edge of the grip with divider points, or other suitable devices. The value of this displacement shall be recorded at failure. This value shall be called the "correction factor."

### Calculations

9. (a) *Breaking Load* shall be calculated by dividing the maximum load in pounds by the original width of the specimen in inches. The result shall be expressed in pounds per inch of width and reported to three significant figures. The thickness of the film shall always be stated to three significant figures (Note 1).

NOTE 1.—This method of reporting is useful for very thin films (0.005 in. and less) for which

breaking load may not be proportional to cross-sectional area and whose thickness may be difficult to determine with precision.

(b) *Tensile Strength* shall be calculated by dividing the maximum (or breaking) load in pounds by the original minimum cross-sectional area of the specimen in square inches. The result shall be expressed in pounds per square inch and reported to three significant figures.

(c) The *corrected* extension shall be calculated by doubling the value of the "correction factor" (Section 8 (g)) and subtracting the result from the extension read from the chart.

(d) *Percentage Elongation* shall be calculated by dividing the corrected extension at the moment of rupture of the specimen by the original distance between grips and multiplying by one hundred. The percentage elongation shall be reported to two significant figures.

(e) *Stress-Strain Data* may be obtained from the load extension chart by selecting points on the recorded curve at equal load intervals, and calculating the corresponding stresses by dividing the loads by the original cross-section. The strain for the selected points on the recorded curve shall be calculated as follows:

(1) The ratio of the *corrected* extension at break as given in Paragraph (c) to the uncorrected extension at break as read from the recorded curve on the chart shall be determined.

(2) By assuming that the ratio of these two extensions is constant at any time during the application of the stress (Note 2), the strain corresponding to the chosen points on the recorded curve shall be calculated in the following manner:

$$\text{Strain} = \frac{(\text{Ext.})_a}{(\text{Ext.})_c} \times \frac{\Delta L_c}{L_c}$$

where:



$\Delta L_c$  = extension between grips taken from the recorded curve for any given load,

(Ext.)<sub>a</sub> = *corrected* extension at break,

(Ext.)<sub>c</sub> = uncorrected extension at break as determined from the recorded curve, and

$L_c$  = original length between grips.

NOTE 2.—It is noted here that this assumption is of questionable validity.

(f) *Elastic Modulus* shall be calculated by drawing a tangent to the initial linear portion of the stress-strain curve, selecting any point on this straight line and dividing the tensile stress (nominal) represented by the point by the corresponding strain (Note 3). The result shall be expressed in pounds per square inch and reported to three significant figures.

NOTE 3.—For the correct method of drawing the tangent to the stress-strain curve and for errors to avoid in this process, see Note 14 in the Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

(g) For each series of tests, the arithmetic mean of all values obtained shall be calculated to three significant figures and reported as the "average value" for the particular property in question.

(h) The deviation of each value from

the "average value" shall be calculated and the arithmetic mean of these deviations determined. This arithmetic mean shall be reported to two significant figures as the "average deviation" of the particular series of results.

## Report

10. The report shall include the following:

(1) Complete identification of the material tested, including type, source, manufacturer's code numbers, form, principal dimensions, previous history, etc.,

(2) Method of preparing test specimens,

(3) Thickness and width of test specimen,

(4) Conditioning procedure used,

(5) Atmospheric conditions in test room,

(6) Number of specimens tested,

(7) Rate of loading,

(8) Rate of stressing as defined in Section 2 (f),

(9) Tensile strength or breaking load, average value and average deviation,

(10) Percentage elongation, average value and average deviation,

(11) Elastic modulus, average value and average deviation, and

(12) Date of test.

# *Tentative Methods of Test for*

## TENSILE AND COMPRESSIVE PROPERTIES OF PLASTICS AT SUBNORMAL AND SUPERNORMAL TEMPERATURES<sup>1</sup>



A.S.T.M. Designation: D 759 - 44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These methods of test are intended for use in determining the tensile and compressive properties of organic plastics at temperatures above and below the standard laboratory temperature.

(b) These methods are not intended specifically for use in determining the effect of long continued exposure at elevated or reduced temperatures or of cyclic exposures to ranges of temperature, although they may be used for such special tests if desired.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Testing Machine*.—The tension testing machine and its accessory equipment shall conform to the requirements specified in the Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638)<sup>3</sup> and the compression testing machine shall con-

form to the requirements specified in the Tentative Method of Test for Compressive Strength of Plastics (A.S.T.M. Designation: D 695).<sup>3</sup> All mechanical parts of the apparatus which are exposed to high or low temperatures during the test shall be adjusted to function normally at these temperatures, and their accuracy shall be verified by calibration tests at the specified test temperatures. Verification shall be repeated as often as necessary to insure correct test readings.

(b) *Insulated Test Chamber*.—An insulated test chamber, through which air is circulated at the desired temperature, to enclose the specimen during the actual test (Note). This chamber shall also enclose at least enough of the testing machine components, such as the tension grips or the compression tool, to insure that conduction of heat through these components will not materially change the temperature of the specimen from that of the test temperature. The temperature of the air in the test chamber may be regulated by any convenient means (Note).

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by Committee E-10 on Standards, January 19, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

NOTE.—For detailed requirements covering enclosures for testing machines and servicing units for maintaining the desired temperature and conditions, reference should be made to the Tentative Specifications for Enclosures for Small Testing Machines for Tests at Subnormal and Supernormal Temperatures of Electrical Insulating Materials and Plastics (A.S.T.M. Designation: D 760)<sup>3</sup> and the Tentative Specifications for Servicing Units for Tests at Subnormal and Supernormal Temperatures of Electrical Insulating Materials and Plastics (A.S.T.M. Designation: D 761).<sup>3</sup>

(c) *Thermocouples or Thermometers.*—Thermocouples or thermometers placed adjacent to or as near as possible to the test specimens in the test and conditioning chambers to indicate the temperature of the air immediately surrounding the specimen.

(d) *Preconditioning Oven.*—A circulation air oven, adjusted at  $50 \pm 3$  C. ( $122 \pm 5$  F.) for preconditioning test specimens.

(e) *Desiccators.*—Desiccators containing anhydrous calcium chloride (or other suitable desiccant) for storage of the preconditioned specimens.

(f) *Conditioning Chamber.*—A chamber for conditioning the specimen at the desired test temperature prior to testing. The conditioning shall be done either in an insulated storage chamber of the circulation air type or in the insulated test chamber. If the specimens are conditioned in the insulated test chamber they shall be placed on a wire-mesh rack or other suitable support during the conditioning period.

### Test Specimens

3. The tension test specimens shall conform to the requirements specified in A.S.T.M. Method D 638. The compression test specimens shall conform to the requirements specified in A.S.T.M. Method D 695.

### Conditioning

4. (a) *Preconditioning.*—All test specimens shall be preconditioned (Note 1) in accordance with Functional Procedure A of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

NOTE 1.—At the higher temperatures, tests will necessarily be on the basis of dried specimens, regardless of the type of preconditioning. Consequently, in order that tests at all temperatures be made on the same basis so far as moisture content is concerned, a preconditioning treatment which removes all moisture for all types of materials is specified.

(b) *Test Conditions.*—The test specimens shall be placed in the insulated conditioning or test chamber, through which air at the desired temperature is circulating, and allowed to remain until in thermal equilibrium with the conditioning atmosphere (Note 2).

NOTE 2.—Suggested minimum conditioning times are 1 hr. for specimens less than 0.25 in. in thickness or 2 hr. for specimens greater than 0.25 in. in thickness.

### Procedure

5. Conditioned samples shall be removed from the conditioning chamber and placed as rapidly as possible in the tension grips or between the compression platens of the testing machine. This entire transfer shall not require longer than 30 sec. After thermal equilibrium has been restored, the specimen shall be tested in tension in accordance with A.S.T.M. Method D 638 or in compression in accordance with the A.S.T.M. Method D 695.

### Report

6. The report shall include the information specified in A.S.T.M. Method

D 638 for tension tests or A.S.T.M. Method D 695 for compression tests, and in addition the following:

(1) Mean temperature in the test enclosure,

(2) Maximum temperature in the test enclosure at any time,

(3) Minimum temperature in the test enclosure at any time,

(4) Average deviation from the mean temperature in the test enclosure during the test, and

(5) Any pertinent information on time of conditioning.

# *Tentative Method for*

## LONG-TIME TENSION TESTS OF PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 674 - 46 T

ISSUED, 1942; REVISED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method covers the determination of the amount of extension of plastics due to the combined effects of tensile stress, time, temperature, and relative humidity, when tested in the form of specimens of standard shape.

(b) The details of this method are largely advisory, since the characteristics of a plastic as to its resistance to extension are not well known. For reference or comparative tests of any given series of materials or specimens, care shall be taken to secure the maximum degree of uniformity in details of preparation, treatment, and handling.

### Significance of Test

2. The engineering significance of data obtained by long-time tension tests is not presently known.

### Definitions

#### 3. (a) *Ultimate (or Tensile) Strength*

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

of a plastic is the tensile load per unit area of original cross-section, expressed in pounds per square inch, required to break a test specimen within the gage boundaries in accordance with the Tentative Method of Test for Tensile Properties of Plastics (A.S.T.M. Designation: D 638).<sup>3</sup>

(b) *Elongation* is the change in length, expressed in inches, produced by a tensile load on a longitudinal section of a test specimen measured between fixed gage points on the specimen.

### Apparatus

4. The apparatus shall consist of the following:

(a) *Testing Machine*.—Any simple suspension device by means of which a predetermined load, accurately measured to within 1 per cent, may be applied in tension as dead weight loading or by the use of a simple lever system. The test system shall be as free from vibration as possible.

(b) *Grips*.—Grips for holding the test

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book

specimens. Grips of the form shown in Fig. 1 are satisfactory. These grips may be made from any noncorroding metal.

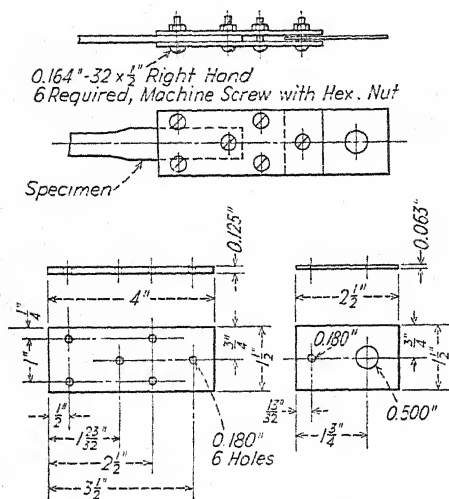


FIG. 1.—Grips for Test Specimen.

(c) *Extensometer*.—A suitable instrument for determining the distance between two fixed points on the test specimen gage length at any time during test. Dividers may be used, since it is essential not to disturb the original position of the specimen to obtain the change in position of the fixed lines on the gage length. A cathetometer or electrical resistance device will yield more accurate results wherever these can be employed.

### Test Specimens

5. (a) The test specimens shall conform to the dimensions shown in Fig. 2. The specimens may be injection or compression molded, or machined from sheets, plates, slabs, or similar material.

(b) When testing materials that may be suspected of anisotropy, duplicate sets of test specimens shall be prepared: one set with the long axis parallel with the direction of anisotropy, and another set with the long axis normal to the direction of anisotropy (Note).

NOTE.—Before testing, all transparent specimens should be given a polariscope inspection and those which show atypical or concentrated strain patterns should be rejected unless these "initial" strains constitute a variable whose effect it is desired to study.

(c) All surfaces of the specimens shall be free from visible flaws, imperfections, or scratches. Marks left by coarse machining operations shall be carefully removed with a fine file, and the filed surfaces shall then be smoothed with abrasive paper (No. 00 or finer). The finishing file strokes or sanding strokes shall be made in a direction parallel with the long axis of the test specimen. All flaws shall be removed from molded specimens, great care being taken not to disturb the molded surfaces.

(d) Gage mark areas should be placed on the specimen with china marking pencil, soft fine crayon, or with India ink. Gage marks may be scratched in the markings, but only when care is taken to avoid injuring the surface of the specimen, since such scratches may cause erroneous results due to the concentrations of stresses.

### Number of Test Specimens

6. (a) At least three specimens shall be tested for each sample in the case of isotropic materials.

(b) Six specimens, three normal to and three parallel with the principal axis of anisotropy, shall be tested for each sample in the case of anisotropic materials.

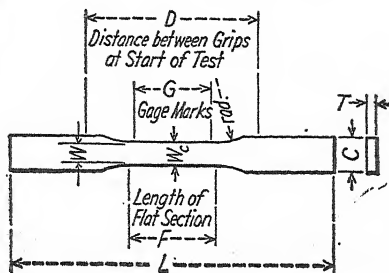
(c) Specimens that fracture at some obvious fortuitous flaw or that do not break between the predetermined gage marks shall be discarded and another specimen substituted.

### Conditioning of Test Specimens

7. (a) Conditioning prior to test shall be in accordance with the Standard Pro-

cedure of the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

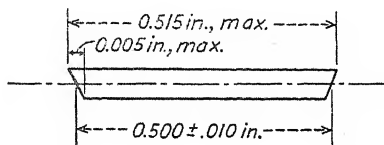
placed in the grips, care being taken not to apply too much tightening pressure but just enough to prevent slippage during the test. The metal flap which is



Dimension, in.	Thickness, $T$ , in. <sup>a</sup>					Tolerances, in.
	$\frac{1}{4}$ in. or under		Over $\frac{1}{4}$ to $\frac{1}{2}$ in., incl.		Over $\frac{1}{2}$ to 1 in., incl.	
	Type I	Type II <sup>e</sup>	Type I	Type II <sup>e</sup>	Type I	
C—Width overall.....	0.750	0.750	1.125	1.125	1.500	$\pm 0.016$
W—Width of flat section <sup>a,b</sup> .....	0.500	0.250	0.750	0.375	1.000	$\pm 0.010$
F—Length of flat section.....	2.25	2.25	2.25	2.25	2.25	$\pm 0.016$
G—Gage length.....	2.00	2.00	2.00	2.00	2.00	$\pm 0.016$
D—Distance between grips <sup>c</sup> .....	$4\frac{1}{2}$	$5\frac{1}{2}$	$4\frac{1}{2}$	$5\frac{1}{2}$	$5\frac{1}{2}$	$\pm \frac{1}{8}$
L—Length overall.....	$8\frac{1}{2}$	$9\frac{1}{2}$	$10\frac{3}{4}$	$10\frac{3}{4}$	12	minimum
Rad.—Radius of fillet.....	3	3	3	3	3	minimum

<sup>a</sup> The width at the center  $W_c$  shall be plus 0.000 in., minus 0.004 in. compared with width  $W$  at other parts of the reduced section. Any reduction in  $W$  at the center shall be gradual, equally on each side so that no abrupt changes in dimension result.

<sup>b</sup> For molded specimens, a draft of not over 0.005 in. may be allowed for the specimens 0.125 in. in thickness, and this should be taken into account when calculating width of the specimen. Thus, the cross-section of the molded specimen would be as follows:



<sup>c</sup> Test marks only.

<sup>d</sup> Thickness,  $T$ , shall be  $0.125 \pm 0.015$  in. for molded specimens and for other specimens where possible. If specimens are machined from sheets or plates, thickness,  $T$ , may be the thickness of the sheet or plate provided this does not exceed 1 in. For sheets of nominal thickness greater than 1 in. the specimens shall be machined to  $1.000 \pm 0.010$  in. in thickness. For sheets of nominal thickness between 1 and 2 in. approximately equal amounts shall be machined from each surface. For thicker sheets both surfaces of the specimen shall be machined and the location of the specimen with reference to the original thickness of the sheet, shall be noted. Tolerances on thickness less than 1 in. shall be those standard for the grade of material tested.

<sup>e</sup> Type I specimens shall be used whenever possible. Type II specimens shall be used only for materials with which the Type I specimen does not give satisfactory breaks in gage length, such as resin-impregnated compressed laminated wood.

FIG. 2.—Tension Test Specimen.

(b) Conditioning during test shall be in accordance with the Standard Laboratory Atmosphere defined in Section 2(a) of A.S.T.M. Methods D 618.

## Procedure

8. (a) The test specimen should be

part of the grip should not be tightened too much in order to enable the load to be axially distributed.

(b) Throughout the duration of the test, observations of extension should be made at sufficiently frequent intervals to define an extension-time curve.



(c) After unloading, a certain amount of prolonged plastic recovery may occur in addition to the elastic recovery. This plastic recovery may be recorded.

(d) The tensile stresses used may be any percentage of the ultimate tensile strength or fixed load agreed upon by the seller and the purchaser.

#### Plotting of Results

9. (a) Extension-time curves may be plotted on semilogarithmic paper with time as the abscissa and unit extension as the ordinate. Many useful relationships may be obtained from such a curve, such as the ratio of the percentage extension to a constant number of hours or the time required to produce a definite percentage extension.

(b) For each series of tests the arithmetic means of all ultimate extension values obtained should be calculated to three significant figures and recorded as the average result for the particular values in question.

(c) The deviation of each value from the average value should be calculated and the arithmetic means of these deviations determined. This value should be recorded to three significant figures as the average deviation of the particular series of results.

#### Report

10. The report shall include the following:

- (1) Date of test,
- (2) Complete identification of the material tested, including type, source, manufacturer's code numbers, form, principal dimensions, and previous history,
- (3) Number of specimens tested,
- (4) Method of preparing test specimens,
- (5) Tensile load applied to test specimen, and
- (6) Average ultimate percentage extension for the period of time used in the test as agreed upon (Section 8 (d)).

## *Tentative Method of Test for* WATER VAPOR PERMEABILITY OF PLASTIC SHEETS<sup>1</sup>



A.S.T.M. Designation: D 697 - 42 T

ISSUED, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test describes procedures for determining the water vapor permeability of plastic sheet made with paper, paperboard, transparent sheeting, plastic sheeting, and other sheet materials suitably bonded together with plastics. There may be considerable difference in the permeability of a plastic sheet when the two faces are exposed, respectively, to (a) a low humidity on one face and a medium humidity on the other, and (b) a high humidity on one face and a medium humidity on the other. These methods provide procedures for the measurement of permeability under both of these conditions. When special use requirements of the materials to be tested are involved, such as applications at other temperatures or under other humidity differences, the test conditions may be varied accordingly.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Test Dish*.—An open mouthed cup or dish of such size and shape that it can be accommodated readily on the pan of an analytical balance. The area of the opening shall be as large as practicable, an area of at least 30 sq. cm. being preferred. The test dish shall be of such design that a satisfactory seal can be made to prevent leakage of water vapor at or through the edges of the test specimen and to define clearly the test area. Suitable designs for the dish with supporting ring or flange are shown in Figs. 1 to 3. Other modifications of these designs may be made, without departing from the principle of preventing edge leakage by means of a complete wax seal.

(b) *Template*.—A template for use in defining the test area and effecting the wax seal. The template shall consist preferably of a circular metal disk  $\frac{1}{8}$  in. in thickness, made from brass, aluminum, or other suitable metal, and having the edge beveled to an angle of about 45 deg. The smaller diameter of the template shall be equal to the diameter of the test area of the specimen.

(c) *Desiccant*.—A desiccant having a powerful affinity for water vapor and a high drying efficiency, that is, a low vapor pressure after absorbing a large

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

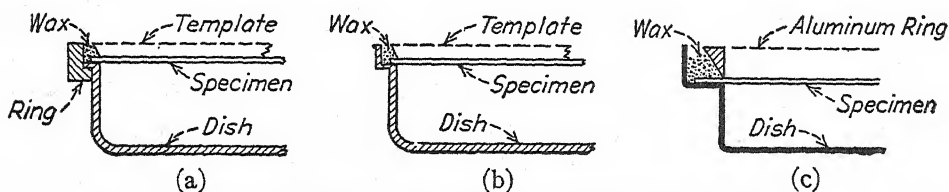
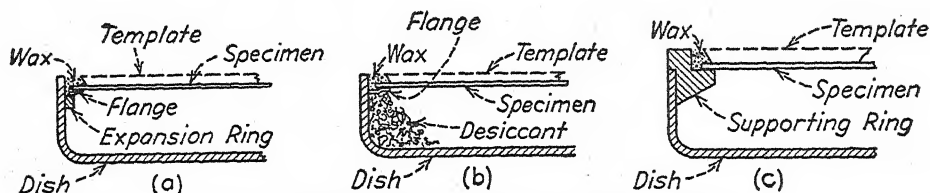
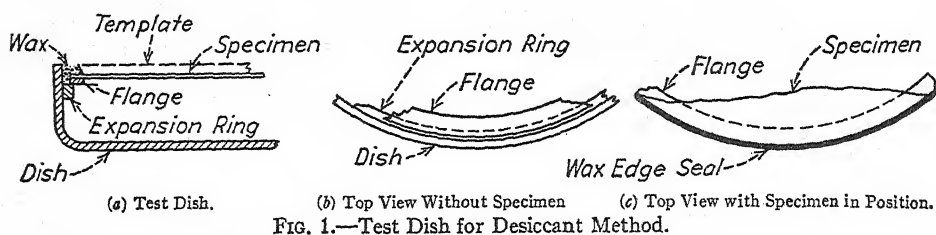
<sup>2</sup> Accepted by Committee E-10 on Standards, August 24, 1942.

amount of water. The desiccant shall remain essentially unchanged in physical condition and exert, while dry, no chemical or physical action on membrane materials with which it is in contact, other than dehydration effects. Anhydrous magnesium perchlorate, or any other desiccant that fulfills these requirements, may be used.

(d) *Wax*.—Wax for sealing the specimen to the test dish. It shall be made

(f) *Balance*.—An analytical balance that has a capacity of 200 g. and is sensitive to 0.001 g.

(g) *Tare Weight*.—A tare weight, for making all weighings on the analytical balance, having a weight about 10 g. less than the assembled test dish and a volume very nearly equal to that of the assembled test dish. The use of this tare weight compensates for any errors in weighing that may arise from changing



of equal parts of crude beeswax and any grade of wood or gum rosin. The wax shall cling tenaciously to surfaces and shall not be brittle at room temperature.

(e) *Petrolatum*.—Petrolatum to be used for application to the beveled edge of the template in order to facilitate removal of the template after sealing the test specimen to the dish. The petroleum jelly commonly used by druggists is suitable for this purpose.

barometric pressure and the consequent change in buoyant effect of the air between successive weighings.

(h) *Test Chamber*.—A testing room or cabinet provided with suitable controls for maintaining a temperature of  $25 \pm 1$  C. ( $77 \pm 2$  F.) at a relative humidity of  $50 \pm 2$  per cent and provided with a fan for maintaining a current of air flowing at a rate of at least 500 ft. per min. over the exposed faces of the speci-

mens under test. A suitable rack shall be provided on which to place the test dishes within the test chamber.

### Test Specimens

3. The test specimens selected from the plastic sheet to be tested shall be representative of the material. The diameter of the specimen shall be equal to the larger diameter of the template. At least two tests shall be made on each side of the sheet, thus requiring at least four test specimens.

#### *Method A. Desiccant Method*

##### Procedure

4. (a) Sufficient desiccant shall be placed in the test dish to cover the test area of the specimen to a depth of at least 15 mm. when the dish is inverted. The specimen shall be placed over the opening of the dish and centered as closely as possible on the supporting ring or flange. With the tip of the finger a thin film of petrolatum shall be applied to the beveled edge of the template. Any petrolatum that may have been deposited on the lower surface of the template shall be wiped off. The template shall be centered exactly over the specimen and dish opening. Molten wax shall be flowed into the annular space surrounding the beveled edge of the template, using a medicine dropper to dispense the molten wax. The template shall be removed from the surface of the specimen as soon as the wax has cooled and solidified.

(b) The assembled test dish shall be weighed on the analytical balance to 0.001 g. using the tare weight. The dish shall be placed on the rack in the test chamber in an inverted position so that the layer of desiccant is in direct contact and evenly distributed over the inner face of the specimen, and so that free access of the conditioned air is pro-

vided on the exposed surface of the specimen.

(c) Successive weighings of the test dish shall be made at intervals of several hours until a constant rate of gain is attained. The increase in weight shall be plotted against the time. The slope of the resulting curve will furnish a measure of the water vapor permeability.

#### *Method B. Water Method*

##### Procedure

5. (a) The use of water inside the dish requires that the test be carried out with the dish in the upright position. As a result, there will exist a layer of air between the surface of the water and the under surface of the specimen through which water vapor will diffuse at a rate that cannot be measured conveniently. Moreover any flange or ring projecting from the wall of the dish will influence the direction of the diffusion of the water vapor. The ratio of water surface area to test area will also have an influence on the water vapor pressure existing on the under surface of the specimen, especially for materials having high permeability. This ratio shall be standardized at 1:1 in accordance with the requirements of Paragraph (b), item (1).

(b) The procedure for making a test by the water method shall be exactly as that described for the desiccant method (See Section 4 (b)) with the following exceptions:

(1) The dish shall be of the type shown in Fig. 3. Its inside diameter shall be at least 60 mm.

(2) Sufficient distilled water shall be placed in the dish to bring the level of the water to such a height that the distance between the surface of the water and the under surface of the specimen is 25 mm. A depth of 5 mm.

of water will adequately take care of losses by evaporation through the most permeable specimens.

(3) The dish shall be placed in an upright position on the rack in the test chamber.

#### REPORT

##### Report

6. The report shall include a detailed summary of the test conditions. The water vapor permeability shall be reported as grams per square meter per 24 hr. at 25 C. (77 F.) and 50 per cent relative humidity, *versus* contact with desiccant or *versus* approximately saturated water vapor derived from a water surface 25 mm. below the specimen. The proper factors shall be used for converting the actual weight change due to permeation of water vapor through the specimen under test, taking into consideration the test area and rate of change in weight. The permeability results shall be reported separately for

each side of the specimen, the opposite sides being designated by a suitable code such as side I and side II. The side of the specimen facing the higher humidity shall be designated in reporting the results. When there is an obvious difference in the two sides of the specimen, there shall be additional identification of the two sides showing this difference, for example, in a specimen waxed on one side only the specimen designation may be "side I, waxed" and "side II, unwaxed".

##### Reproducibility of Results

7. Duplicate determinations should check within plus or minus 10 per cent, depending largely on the variation in the plastic sheet. Very permeable plastic sheet may have a permeability 1000 times greater than a plastic sheet of low permeability. A precision of plus or minus 10 per cent on a given plastic sheet will, therefore, establish it quite definitely in the scale of permeabilities normally encountered in plastic sheet.

# *Tentative Recommended Practice for*

## ACCELERATED WEATHERING OF PLASTICS USING S-1 BULB AND FOG CHAMBER<sup>1</sup>



A.S.T.M. Designation: D 795 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This recommended practice is intended to define conditions for the exposure of plastic materials to artificial sunlight and fog.

NOTE.—Some correlation has been observed between the changes in certain physical properties of certain plastics resulting from exposure to the conditions of this recommended practice and those resulting from a much longer exposure out-of-doors in Washington, D. C., but no general assumptions as to the existence or the extent of such correlation should be made.

(b) This recommended practice is limited to the method of obtaining the exposure conditions, the type of sample, and the procedure to be followed, and does not cover methods of test to be used in evaluating the effects of the exposure.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Lamp*.—A General Electric sunlamp, model BM-12, or an equivalent lamp, equipped with an oxidized alu-

minum reflector approximately 15 in. in diameter at the lower rim, and an S-1 bulb which has been in use at least 50 and less than 550 hr. The S-1 bulb consists of a combination tungsten filament-mercury arc enclosed in Corex D glass which absorbs most of the ultraviolet radiation below 2800 Å (Note 1). The bulb is rated at 400 w. The operating voltage shall be maintained at 110 ± 2 v. (Note 2).

NOTE 1.—To measure the ultraviolet light output of the S-1 bulb, the uranyl-oxalate actinometer may be used. This method is described in the Tentative Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (A.S.T.M. Designation: D 749).<sup>3</sup> As no minimum value for the ultraviolet output has been established, actinometer data are useful only for comparisons between lamps.

NOTE 2.—To control voltage within the limits specified, an automatic voltage stabilizer, such as General Electric stabilizer, Catalog No. 68G158, may be used.

(b) *Disk*.—A phonograph turntable, operating at approximately 33 rpm., on which shall be mounted a light-colored corrosion-resistant metal disk approxi-

<sup>1</sup> Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

mately 17 in. in diameter and 0.1 in. in thickness. Two sets of 21 corrosion-resistant machine screws and nuts shall be attached to the disk at holes equally spaced on concentric circles of approximately  $3\frac{3}{4}$ -in. and 8-in. radii, respectively. A third set of 21 machine screws and nuts shall be attached to the disk at holes spaced midway between the lines connecting the two previous sets of

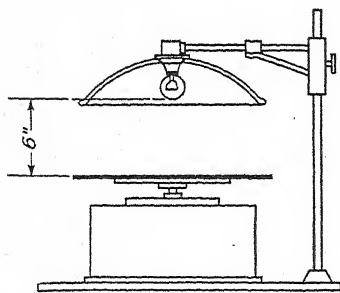
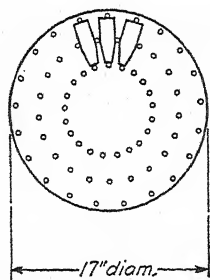


FIG. 1.—Phonograph Turntable and Disk Showing Assembly with Lamp.

screws and on a circle of approximately 6-in. radius, concentric with the other circles. The screws may be American Standard No. 10, 32 threads to the inch,  $\frac{1}{2}$  in. in length with fillister head. Corrosion-resistant washers about  $\frac{5}{8}$  in. in diameter and 0.05 in. in thickness shall be placed over the nuts to support the specimens about  $\frac{3}{16}$  in. above the surface of the disk. (See Fig. 1.)

(c) *Thermometer*.—An A.S.T.M. Partial Immersion Thermometer having a

range of  $-20$  to  $+150$  C. and conforming to the requirements of thermometer 1C - 39 as prescribed in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1).<sup>3</sup>

(d) *Fog Chamber*.—A closed shallow box fitted with a spraying unit and a baffle to prevent the spray from impinging directly on the specimens. The distilled water spray should deposit a fine mist on the specimens without washing action. The temperature of the water feeding the spray shall be 21 to 27 C. (70 to 80 F.).

NOTE.—A box and spray assembly constructed as shown in Figs. 2 and 3 is suitable.

### Test Specimens

3. The specimens shall be 4 in. in length and  $1\frac{1}{2}$  in. in width with a tapered end as shown in Fig. 4 to permit assembly of 21 specimens on the disk at one time.

### Procedure

4. (a) The specimens shall be placed on the disk so that they are supported above its surface by the washers. The disk shall be centered under the S-1 bulb so that the bottom plane of the specimens is 6 in. from the bottom of the bulb.

(b) The test shall be so conducted that with the thermometer placed on the turntable with its midpoint at the center of the disk, it will read 55 to 60 C. (Note 1). If during the test the thermometer reads higher than 60 C. a fan shall be provided, placed so that it blows against the bottom of the turntable a volume of air sufficient to bring the thermometer reading to 55 to 60 C. (Note 2).

NOTE 1.—The thermometer does not necessarily indicate the temperature of the specimen or of the air in the plane of the specimen, since the reading of the thermometer is determined partly by the amount of radiant energy which it absorbs and partly by the temperature of its surroundings.



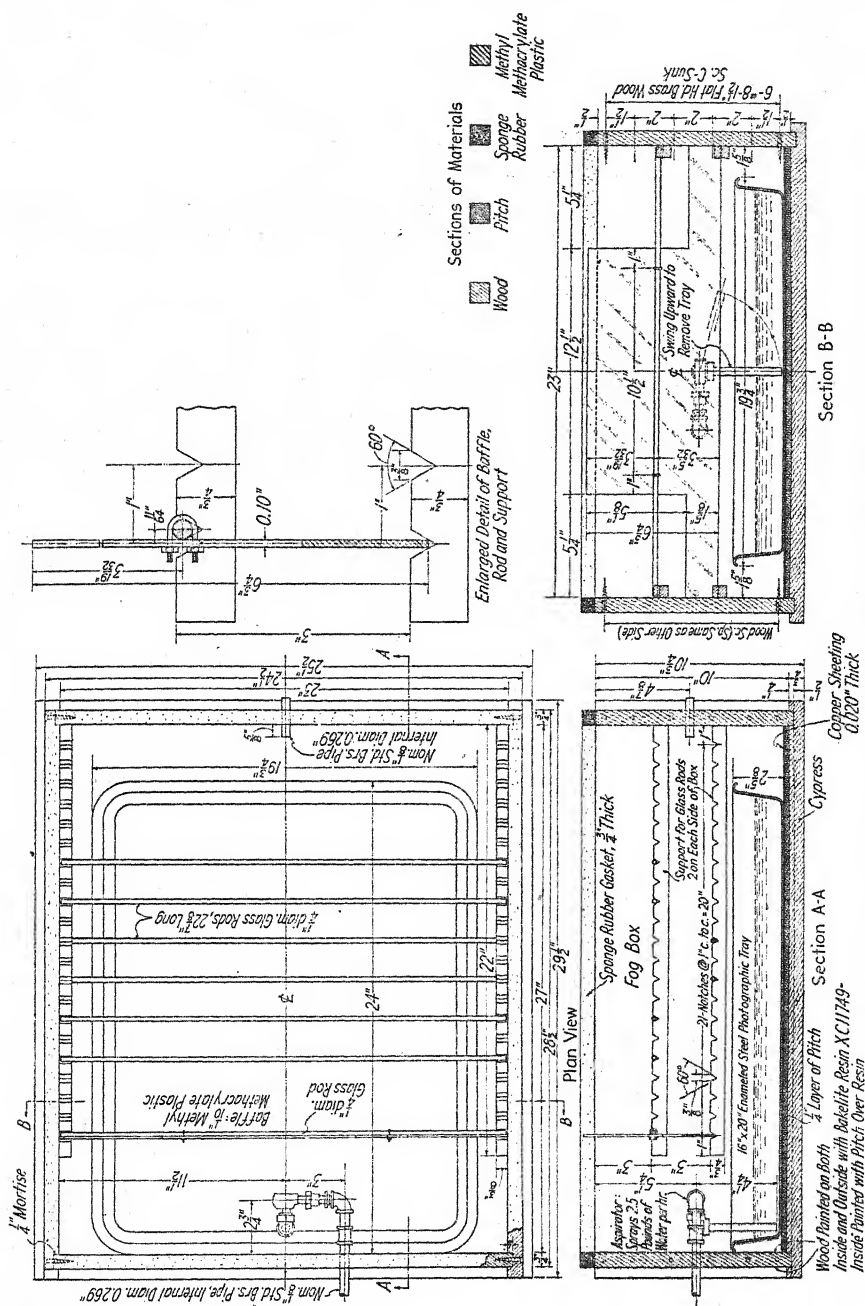


FIG. 2.—Fog Box and Spray Assembly for Weathering Test.

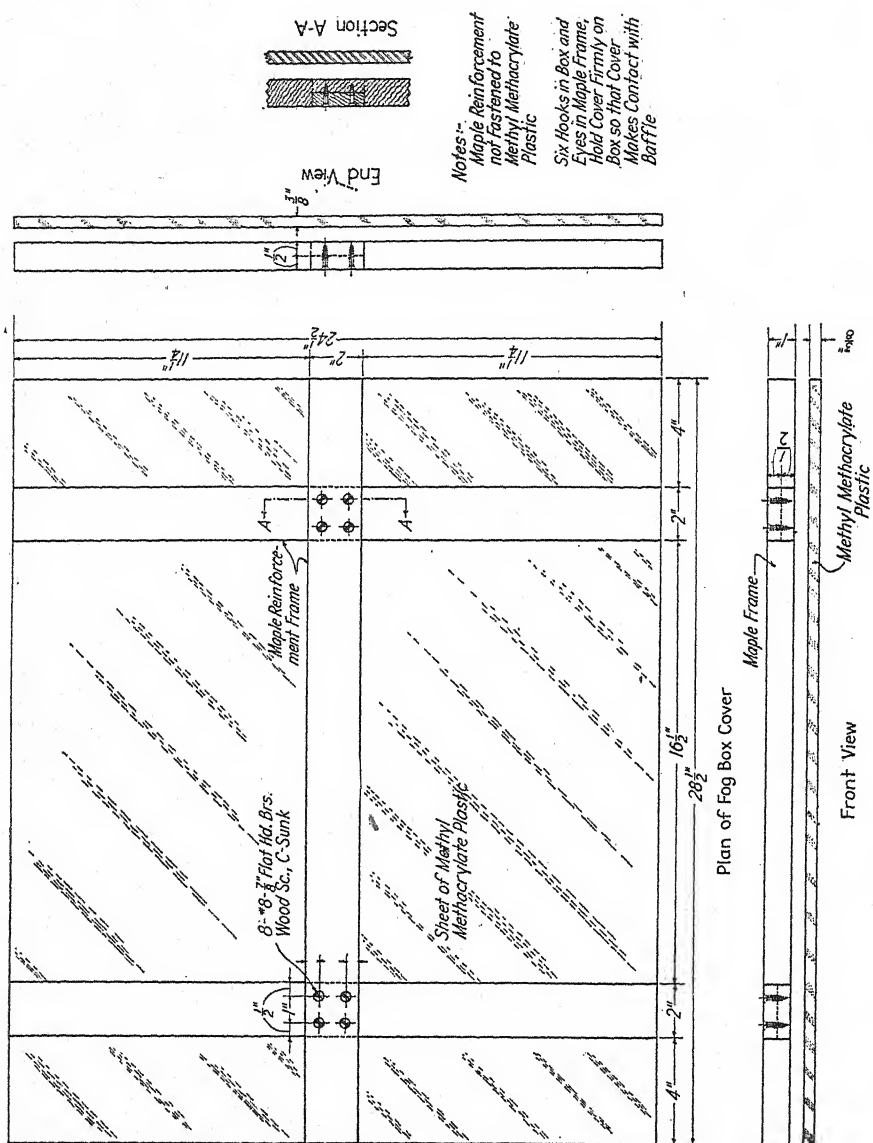


FIG. 3.—Details of Fog Box Cover and Frame.

NOTE 2.—If the apparatus is not enclosed, and operates in a room in which the ambient temperature is 70 to 80 F., the thermometer reading will lie between 55 and 60 C. If the reading is below 55 C., the temperature can be raised by reducing somewhat the air circulation around the lamp.

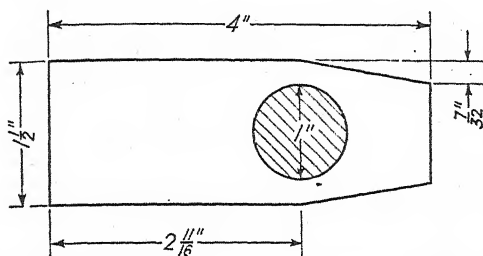


FIG. 4.—Test Specimen for Weathering Test.

(c) Radiation and wetting cycles for each 24-hr. period shall be in the following order: 2 hr. in the fog box, 2 hr. irradiation, 2 hr. in the fog box, and 18 hr. irradiation. The initial period may be any of these, according to convenience.

(d) The specimens shall always be exposed throughout the test with the same side up, and before making any tests the

specimens shall be wiped with a damp cloth to remove any adhering dirt. The area exposed between the concentric circles of 3 3/4 and 6-in. radii, respectively, shall be used to determine the effects of the test conditions on the specimens. The duration of the accelerated weathering test shall be 240 hr., including the time of exposure to fog, unless otherwise specified.

### Report

5. The report shall include the following:

(1) Duration of the test in hours including exposure to fog.

(2) Observations regarding surface changes (such as color, dulling, and chalking) and deep-seated changes (such as checking, crazing, warping, and discoloration).

(3) The results of any physical or chemical tests made to determine the extent of degradation resulting from the exposure. The test methods used shall be adequately described.

## *Tentative Method of Test for*

### IMPACT RESISTANCE OF PLASTICS AT SUBNORMAL AND SUPERNORMAL TEMPERATURES<sup>1</sup>



A.S.T.M. Designation: D 758 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. This method of test is intended for use in determining impact resistance of organic plastics under conditions of low moisture content at temperatures above and below the standard laboratory temperature.

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Testing Machine*.—The testing machine and its accessory equipment shall conform to the requirements specified in the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256).<sup>3</sup> For tests at low temperatures, care shall be taken that the testing machine operates in the usual manner. This may be accomplished by washing out the oil used as a lubricant and replacing it with kerosine. A change of not greater than 50 per cent in the

windage and friction corrections is permissible.

(b) *Insulated Test Chamber*.—An insulated test chamber, through which air is circulated at the desired temperature, to enclose the entire testing machine during the test (Note). The chamber shall be equipped with a window through which may be read the scale and the thermometer near the specimen, and a door for inserting the specimens. Means shall be provided outside the test chamber for tripping the hammer of the impact machine. In the case of cantilever beam (Izod type) impact tests, external means shall also be provided for tightening the vise (see Note, Section 5). The equipment shall be capable of maintaining the test temperature within plus or minus 2 C. of the specified temperature at the location of the thermometer near the test specimen, and a difference in temperature between the inlet and outlet duct thermometers of not greater than 5 C. at equilibrium.

NOTE.—For detailed requirements covering enclosures for testing machines and servicing units for maintaining the desired temperature and conditions, reference should be made to the

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by Committee E-10 on Standards, January 19, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

Tentative Specifications for Enclosures for Small Testing Machines for Tests at Subnormal and Supernormal Temperatures of Electrical Insulating Materials and Plastics (A.S.T.M. Designation: D 760)<sup>3</sup> and the Tentative Specifications for Servicing Units for Tests at Subnormal and Supernormal Temperatures of Electrical Insulating Materials and Plastics (A.S.T.M. Designation: D 761).<sup>3</sup>

(c) *Thermometers*.—Three thermometers, one each at the inlet and outlet ducts of the chamber, and another placed as close as possible to the specimen but in no case greater than 3 in. from the specimen.

(d) *Preconditioning Oven*.—A circulation air oven, adjusted at  $50 \pm 3$  C. ( $122 \pm 5$  F.) for preconditioning test specimens.

(e) *Desiccators*.—Desiccators containing anhydrous calcium chloride (or other suitable desiccant) for storage of the preconditioned specimens.

(f) *Conditioning Chamber*.—A chamber for conditioning the specimen at the desired test temperature prior to testing. The conditioning shall be done either in an insulated storage chamber of the circulation air type or in the insulated test chamber. If the specimens are conditioned in the insulated test chamber they shall be placed on a wire-mesh rack or other suitable support during the conditioning period.

### Test Specimens

3. The test specimens shall conform to the requirements specified in A.S.T.M. Methods D 256.<sup>3</sup>

### Conditioning

4. (a) *Preconditioning*.—All test specimens shall be preconditioned in accordance with Functional Procedure A of the Tentative Methods of Conditioning

Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618).<sup>3</sup>

(b) *Test Conditions*.—Test specimens shall be placed in the conditioning chamber and subjected to the test temperature in dry circulating air for at least 1 hr. This temperature shall be controlled within plus or minus 2 C. of the specified test temperature.

### Procedure

5. Conditioned specimens shall be removed from the conditioning chamber (Section 4 (b)) and placed as rapidly as possible in the testing machine. The entire transfer shall not require more than 30 sec. After thermal equilibrium has been restored but in no case in less than 5 min., the air circulation shall be cut off, the vise tightened (in the case of cantilever beam (Izod type) impact tests, see Note), and the hammer of the impact machine tripped. In other respects the test procedure shall be in accordance with that prescribed in A.S.T.M. Methods D 256.<sup>3</sup>

NOTE.—During the 5 min. or more between the closing of the test chamber and the tripping of the hammer, the test specimen is under pressure from the vise. At high temperatures, this may cause some materials to flow sufficiently to become loosened. It is, therefore, necessary to give the vise a final tightening from outside just before the actual impact.

### Report

6. The report shall include the information specified in A.S.T.M. Methods D 256<sup>3</sup> and in addition the following:

(1) Readings of the three thermometers just before the test, and

(2) Any pertinent information on the time of conditioning.

# *Tentative Recommended Practice for* MOLDING SPECIMENS OF PHENOLIC MATERIALS<sup>1</sup>



**A.S.T.M. Designation: D 796 - 45 T**

ISSUED, 1944; REVISED, 1945.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. This recommended practice covers procedures for molding test specimens of phenolic materials, as follows:

*General Purpose Phenolic Materials.*—

Impact, flexure, and tension test specimens.

*Shock-Resistant Phenolic Materials.*—

Impact, flexure, and tension test specimens.

*Mica-Filled Phenolic Materials.*—Im-

pact, flexure, and tension test specimens.

## Apparatus

2. The apparatus shall consist of the following:

(a) *Molds:*

(1) For molding impact and flexure specimens of general purpose mica-filled and shock-resistant phenolic materials, the single bar, single cavity positive mold described in Fig.

1 of the Tentative Specifications for Molds for Test Specimens of Molding Materials Used for Electrical Insulation (A.S.T.M. Designation: D 647)<sup>3</sup> shall be used.

(2) For molding tension specimens of general purpose, mica-filled and shock-resistant phenolic molding materials, the positive mold shown in Fig. 3 of A.S.T.M. Specifications D 647 shall be used.

(b) *Press.*—A hydraulic press of sufficient ram diameter so that the molding pressure on the specimen can be controlled to 250 psi.

(c) *Heating System.*—Any convenient method of heating the press platens or molds may be used provided the heat source is constant enough to maintain the mold temperature within plus or minus 3 C. (5 F.).

(d) *Thermometer.*—A 1½-in. immersion mercury thermometer having a diameter just under  $\frac{3}{16}$  in. and a temperature scale of not more than 20 C. per in. of

<sup>1</sup> Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE I.—MOLDING CONDITIONS FOR SPECIMENS OF PHENOLIC MATERIALS.

Molding Conditions	General Purpose Phenolic Materials	Mica-Filled Phenolic Materials	Shock-Resistant Phenolic Materials
	Impact, Flexure, and Tension Specimens	Impact, Flexure, and Tension Specimens	Impact, Flexure, and Tension Specimens
Molding pressure, psi. ....	2500 ± 500	2500 ± 500	4000 ± 500
Molding temperature. ....	160 ± 3 C. (320 ± 5 F.)	150 ± 3 C. (300 ± 5 F.)	155 ± 3 C. (310 ± 5 F.)
Charge. ....	powder	powder	powder
Cure. ....	5 min.	10 min.	10 min.
Breathing mold. ....	Breathing the mold may be necessary for molding most materials of these types.		

length or a mold pyrometer graduated to read 5 C.

### Conditioning

3. Materials shall be used in the as-received condition. For specimens to be used in referee tests, the materials shall

be conditioned for 72 hr. in a desiccator over anhydrous calcium chloride at room temperature. The material shall be spread to a depth of not more than  $\frac{1}{4}$  in.

### Procedure

4. Test specimens shall be molded under the conditions prescribed in Table I.



# *Tentative Recommended Practice for*

## DETERMINING PERMANENT EFFECT OF HEAT ON PLASTICS<sub>1</sub>



A.S.T.M. Designation: D 794 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This recommended practice is intended to define the conditions for testing the resistance of plastic sheet, plastic laminated materials, and molded plastics to permanent changes in properties due to exposure at elevated temperatures. Only the method of heat exposure is specified, and not the specimen; so that the permanent effect of heat on any property may be determined by use of the proper specimen.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Circulation Air Oven*.—A circulation air oven capable of maintaining the temperature within plus or minus 2 C. of the specified test temperature.

(b) *Testing Device*.—Testing devices for determining specific properties before and after exposure to heat shall conform to the requirements prescribed in the method of test of the American Society for Testing Materials covering the specific property to be determined.

### Test Specimens

3. The number and type of test specimen required shall be in accordance with the A.S.T.M. test method for the specific property to be determined.

### Determination of Properties Prior to Heat Exposure

4. Initial tests shall be conducted in the Standard Laboratory Atmosphere as specified in the Tentative Methods of Conditioning Plastics and Electrical Insulating Materials for Testing (A.S.T.M. Designation: D 618),<sup>3</sup> and in accordance with the requirements of the A.S.T.M. test method for determining the specific property or properties required.

### Exposure of Specimens for Heat Effect

5. Test specimens shall be suspended from one end in the circulation air oven so that free access to air shall be assured to all sides. At the conclusion of the exposure period, the specimens shall be removed from the oven, conditioned, and tested as specified in Section 4. Individual specimens shall be exposed at one temperature only. Each new exposure

<sup>1</sup> Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

temperature shall require additional, properly conditioned specimens.

#### Heat Exposure Schedule

6. (a) *Selection of Approximate Temperature at Which Failure Occurs.*—For a rapid evaluation of the effect of heat and the establishment of the approximate temperature range for a more critical study, the following schedule shall be observed:

(1) The starting temperature shall be a multiple of 25 C. which, experience has indicated, is near the maximum which the plastic will stand without failure, for example, 25, 50, 75, 100, 125, 150, 175, and 200 C.

(2) The period of exposure to heat shall be 4 hr.

(3) Exposure temperatures shall be increased in steps of 25 C. until failure occurs. Should failure occur at the temperature first chosen under Item 1, the exposure temperature shall be decreased in steps of 25 C. to that point at which failure does not take place.

(4) Failure is defined as a change in physical appearance, weight, length, or other properties in excess of the agreed limits.

(b) *Determination of Heat Resistance.*

—The following schedule shall be observed for determining the maximum

temperature at which the plastic is resistant:

(1) The starting temperature shall be the nearest multiple of 10 C., at least 30 C. below that selected under Paragraph (a); for example, if failure occurs at 150 C. in Paragraph (a), then the starting temperature will be 120 C. If failure occurs at 175 C., the starting temperature will be 140 C.

(2) Specimens shall be exposed for a period of 7 days.

(3) Subsequent exposure temperatures shall be increased or decreased in steps of 10 C. until a maximum is determined above which permanent change occurs in the property being tested greater than the agreed limits.

#### Report

7. The report shall include the following:

(1) The property or properties on which the effect of heat was determined,

(2) The agreed tolerance in property change,

(3) The maximum temperature to the nearest 5 C. to which the plastic was exposed under the conditions of this test with a change in property not exceeding the agreed limit, and

(4) Observations on any visible changes in the test specimen.

# *Tentative Definitions of*

## TERMS RELATING TO PLASTICS<sup>1</sup>



A.S.T.M. Designation: D 883 - 46 T

ISSUED, 1946; REVISED, 1946.<sup>2</sup>

These Tentative Definitions have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Plastic, *n.***—A plastic is any one of a large and varied group of materials which consists of, or contains as an essential ingredient, an organic substance of large molecular weight and which, while solid in the finished state, at some stage in its manufacture has been or can be formed (cast, calendered, extruded, molded, etc.) into various shapes by flow, usually through the application singly or together of heat and pressure.

**Plastic, *adj.***—The adjective plastic indicates that the noun modified is made of, consists of, or pertains to plastic.

<sup>1</sup> Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee D-20 on Plastics.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946. Revision accepted by the Administrative Committee on Standards, July 10, 1946.

**NOTE.**—The above definition may be used as a separate meaning to the definitions contained in the dictionary for the adjective "plastic."

**Pseudo-stable, *adj.***—Pseudo-stable denotes an unstable condition of a plastic under constant environment during which time it is rapidly changing its physical properties.

**NOTE.**—Pseudo-stable refers, for example, to the temporarily more flexible condition of some plastics after molding. No physical tests should be made while the plastic is in a pseudo-stable condition unless data regarding this condition are desired.

# *Tentative Methods of*

## CHEMICAL ANALYSIS OF RUBBER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 297 - 43 T

ISSUED, 1940; REVISED, 1941, 1943.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods of chemical analysis of rubber products are classified for the purpose of this standard into the following five separate procedures:

- A. Complete Procedure,
- B. Rubber Solvent Method,
- C. Short Procedure,
- D. Direct Determination of Rubber Hydrocarbon, and
- E. Copper and Manganese Determination in Crude Rubber.

Method A is complete and of general application; it includes methods for the chemical analysis of both soft and hard rubber compounds. Method B employs a solvent for the separation of vulcanized rubber from compounding ingredients in order to avoid thermal decomposition. Method C is designed

to permit rapid analysis of specific types of rubber compounds. Method D is a procedure for the direct determination of rubber hydrocarbons by quantitative oxidation with chromic acid and measurement of the acetic acid thereby produced instead of employing the usual estimation by difference. Method E includes specialized procedures for the determination of copper and manganese in crude rubber only.

### Description of Terms

2. (a) *Acetone Extract*.—If the acetone extraction is made on vulcanized compounds, the acetone removes the rubber resins, the free sulfur, any mineral oils or waxes, any acetone-soluble antioxidants and organic accelerators or their decomposition products, and part of any bituminous substances or vulcanized oils that may have been used. This is generally called acetone extract uncorrected. The percentage of free sulfur and the percentage of waxy hydrocarbons are determined and their sum deducted from the total extract. The value obtained is known as acetone

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

These methods are essentially those adopted as standard by the American Chemical Society and the National Bureau of Standards and include the Joint Rubber Insulation Committee's Procedure.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, November 20, 1943.

Prior to their present publication as tentative, these methods were published as tentative from 1928 to 1932. They were adopted in 1932, published as standard from 1932 to 1940, being revised in 1936, 1938, and 1939, but withdrawn, revised, and republished as tentative in 1940.

extract, corrected. The corrected figure thus obtained will at times give valuable information regarding the quality of the rubber present. This is not true, however, when the compound contains substantial quantities of mineral oils or waxes, bituminous substances, organic accelerators, or antioxidants which have been added. With compounds containing rubber value which consists of only the best grades of Hevea rubber, the acetone extract should not exceed 5 per cent of the rubber present. A higher extract may indicate the presence of inferior or reclaimed rubbers, added oils, waxes, or bituminous materials, or substantial quantities of organic accelerators or antioxidants. No correction is possible for small quantities of antioxidants and organic accelerators, since no general method is now known for the separation and identification of all classes of these materials.

(b) *Chloroform Extract*.—The chloroform extraction removes a portion of the bituminous substances and serves as an indication of their presence. The chloroform extract may also include other materials.

(c) *Alcoholic-Potash Extract*.—The purpose of the alcoholic-potash extraction is to detect the presence of rubber substitutes.

(d) *Free Sulfur*.—Free sulfur is that which is removed during the acetone extraction.

(e) *Total Sulfur*.—Total sulfur is the sulfur that occurs in the compound, either free or chemically combined, exclusive of that sulfur which may be present in barium sulfate.

(f) *Ash*.—The ash is the residue left after ignition and consists principally of the nonvolatile mineral fillers, together with their reaction products with sulfur.

(g) *Sulfur in Ash*.—The sulfur in ash consists of the sulfur from the mineral fillers and also part of the sulfur that was with the rubber, but which during

ignition enters into combination with mineral fillers.

(h) *Total Compounding Ingredients*.—Total compounding ingredients consist of the residue obtained when the rubber is dissolved by mineral oil in the solution method of analysis. Total compounding ingredients corrected is the value obtained after deducting sulfur and organic matter present.

(i) *Total Extract*.—The total extract is the material removed from the rubber compound by extraction with a mixture consisting of 32 per cent acetone and 68 per cent chloroform by volume, for a period of at least 8 hr.

### Reagents

3. (a) *General Requirements*.—All ordinary reagents used shall be of the "Reagent Grade."

(b) *Acetone*.—Distill acetone of U.S.P. purity over anhydrous potassium carbonate, not more than 10 days before use, using the fraction which distills at 56 to 57 C.

(c) *Alcoholic Potash Solution*.—Prepare a 1 N alcoholic potash solution by dissolving the required amount of KOH in alcohol that has been purified as follows: Dissolve 1.5 g. of  $\text{AgNO}_3$  in 3 ml. of water and add it to 1000 ml. of alcohol. Dissolve 3 g. of KOH in the smallest amount of hot water, cool, add it to the alcoholic-silver nitrate solution, and shake thoroughly. Allow the solution to stand for at least 24 hr., filter, and distill. (Ethyl alcohol denatured with 10 per cent by volume of methanol may be used in place of pure ethyl alcohol.)

(d) *Nitric Acid - Bromine Solution*.—Add a considerable excess of bromine to  $\text{HNO}_3$  (sp. gr. 1.42) so that a layer of bromine is present in the reagent bottle. Shake thoroughly and allow to stand 24 hr. before using.

(e) *Zinc - Nitric Acid Solution*.—Add 200 g. of zinc oxide to 1000 ml. of  $\text{HNO}_3$  (sp. gr. 1.42).

(f) *Barium Chloride Solution* (100 g. per l.).—Dissolve 100 g. of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  in 1 liter of distilled water, and add two to three drops of  $\text{HCl}$  (sp. gr. 1.19). If there is any insoluble matter or cloudiness, heat the solution overnight on a steam bath, and filter.

(g) *Standard Potassium Bromate Solution* (0.1 N).—Dissolve approximately 2.79 g. of  $\text{KBrO}_3$  in 1 liter of water (Note). Standardize this solution by means of standard arsenious oxide as follows. Use a sample containing from 0.1 to 0.2 g. of  $\text{As}_2\text{O}_3$ . Dissolve it in  $\text{KOH}$ , neutralize with  $\text{HCl}$ , and add 15 ml. of  $\text{HCl}$  (sp. gr. 1.19) in excess. Dilute to 100 ml., warm to about 60 C., and titrate with  $\text{KBrO}_3$  using two drops of methyl red solution (0.2 per cent) as indicator. When the indicator fades, add the  $\text{KBrO}_3$  slowly, using more indicator if desired. At the end point the solution turns colorless and an added drop of indicator should be decolorized.

Sb equivalent of 1 ml. of  $\text{KBrO}_3$  =  

$$\frac{\text{wt. of } \text{As}_2\text{O}_3 \times 1.23}{\text{milliliters of } \text{KBrO}_3}$$

NOTE.—Potassium bromate of known purity may be used as a primary standard for making this solution. Both the solid and the solution are very stable. If  $\text{KBrO}_3$  is used as a primary standard, 1 ml. of 0.1 N  $\text{KBrO}_3$  is equivalent to 0.006089 g. of antimony.

(h) *Starch Iodate Paper*.—Impregnate filter paper with a solution obtained by heating 2 g. of starch with 100 ml. of water and, after solution, add 0.2 g. of potassium iodate dissolved in 5 ml. of water.

(i) *Rubber Solvent*.—The mineral oil used in the solution method of determining rubber and fillers shall have approximately the following properties:

Saybolt	
Universal viscosity	{ at 68 F. (20 C.) . . 56 sec. at 100 F. (38 C.) . . 45 sec.
Flash point.....	270 F. (132 C.)
Fire point.....	350 F. (177 C.)
Specific gravity.....	0.853
Color.....	colorless

(j) *Asbestos Gooch Crucible*.—Prepare a Gooch crucible in the following manner: Cut some amphibole asbestos into fine pieces with shears, digest with  $\text{NaOH}$  (10 per cent), wash with water and then digest with  $\text{HCl}$  (sp. gr. 1.19) for a few hours on a steam bath. After it has been washed comparatively free from acid by decantation, shake the asbestos up with water and use the resulting mixture in preparing the pad. After igniting the Gooch crucible, it is then ready for use.

(k) *Chloroform*.—Chloroform of U.S.P. purity shall be used in extraction.

### Blank Determinations

4. Blanks shall be run on all determinations to check the purity of the materials used and deductions shall be made accordingly.

### Check Determinations

5. In the event of any determination not falling within limits where given in these methods of test, duplicate check determinations shall be made, the average of which shall fall within the limits specified and shall be taken as the true value.

### Preparation of Samples

6. (a) Before preparing a sample for analysis, the analyst shall, by inspection, assure himself that it has not been contaminated. The sample to be analyzed shall be selected by taking pieces from various parts of the original sample and separating them from foreign matter. Because of the variety of rubber products to which this method is to be applied, no single procedure for reducing the sample to the required fineness is applicable to all samples. Furthermore, the best methods usually require the use of expensive apparatus. Therefore, several alternative procedures for this purpose are described in the following Paragraphs (b) to (g). The analyst is

expected to select the one most suitable to the sample that he is analyzing and the equipment available.

(b) For vulcanized soft rubber, unvulcanized rubber, crude rubber, and many samples of reclaimed rubber, it is preferable to mix the sample and grind it by passing it two or three times through a clean, cold laboratory rubber mill. The rubber will come from the mill in the form of a coarse powder or a rough sheet. If the product is in the form of a sheet, the adjustment of the mill shall be such that the thickness of the final sheet is no greater than 0.5 mm. If the sample is sticky, it shall be rolled in holland cloth for storage preliminary to the final sampling for analysis. If the milled sample is a powder, it shall be transferred to a 1410-micron (No. 14) sieve<sup>3</sup> and rubbed through the sieve. Grinding shall be continued until the entire sample passes through the sieve.

(c) In the absence of milling machinery, the sample may be prepared by cutting it with scissors so that it will pass a 1410-micron (No. 14) sieve.<sup>3</sup> The sample may be cut into long strips that are fine enough to pass freely through the sieve and the strips fed through by hand, or the sample may be cut into small fragments and shaken through the sieve. The cutting shall be continued until the entire sample passes through the sieve. If necessary, to prevent sticking, different fragments of the sieved sample may be segregated by wrapping in holland or other cotton cloth.

(d) Certain very glutinous samples may be prepared for extraction analysis as follows: Place a weighed 2-g. sample of the material between two pieces of ashless filter paper that have been ex-

tracted according to the procedure described in Section 10 (b). The papers should be approximately 20 by 4 in. and the sample should be placed near one end. Flatten the sample and spread it throughout the length of the filter paper by passing the "sandwich," lengthwise, through a cold, closely set, even-speed rubber calender. The gross thickness of the resulting sheet should not be greater than 1.0 mm. If a rubber calender is not available, a similar sheet may be obtained by placing the sample in a hydraulic press or a vise. In the latter case, the sample may be roughly spread by hand throughout the length of the filter paper and pressure applied to small areas at a time until the whole sample has been flattened.

(e) Samples of rubberized cloth, whose over-all thickness is no greater than 1.0 mm., may be prepared for analysis by cutting them into pieces 1.5-mm. square, and then mixing well. If the fabric is easily removed, it should be separated unless an analysis of the whole cloth is desired.

(f) Samples of rubber cements shall be evaporated to dryness in a vacuum at a temperature not higher than 30 C. The residue may then be analyzed as an unvulcanized sample. A separate sample of the cement shall be distilled under reduced pressure if examination of the solvent is desired.

(g) Samples of hard rubber shall be reduced to powder form by filing, cleaned with a magnet, and sieved through a 590-micron (No. 30) sieve.<sup>3</sup> Residue retained on this sieve shall be reduced until the entire sample passes through the sieve.

#### Preliminary Examination of Sample

7. (a) *Carbonates*.—Drop a small piece of the sample into a test tube containing HCl (sp. gr. 1.19) saturated

<sup>3</sup> Detailed requirements for these sieves are given in the Standard Specifications for Sieves for Testing Purposes (A.S.T.M. Designation: E 11), see p. 471.



with bromine. If a stream of bubbles is given off, the Rubber Solvent Method (Note) described in Sections 35 to 39, inclusive, shall be used.

(b) *Antimony*.—Place a portion of the sample in a porcelain crucible and burn off the rubber. Treat the ash with concentrated HCl (sp. gr. 1.19). Dilute the solution to ten times its volume, filter, and saturate with  $H_2S$ . If a precipitate of antimony sulfide is formed, the method described in Section 21, or the Rubber Solvent Method (Note) in Sections 35 to 39, inclusive, shall be used.

(c) *Carbon Black*.—Heat a portion of the sample with  $HNO_3$  (sp. gr. 1.42) until there is no more frothing. If the liquid is black, it indicates the presence of free carbon, and the test for this substance should be made.

(d) *Barium Salts*.—A qualitative test for barium can conveniently be made on the residue after filtration in the total sulfur determination as described in Section 15, by dissolving it in HCl (10 per cent), filtering, and then adding a few drops of  $H_2SO_4$  (10 per cent). A white precipitate of barium sulfate indicates the presence of barium in the sample.

(e) *Paraffin*.—If paraffin is present, it will solidify in the cold acetone extract as a white flocculent precipitate clinging to the sides of the flask.

(f) *Glue*.—Extract a portion of the sample with a mixture of 32 per cent acetone and 68 per cent chloroform by volume for 8 hr. Dry the sample and digest for 1 hr. with hot water. Filter, cool, and add a few drops of a freshly prepared solution of tannic acid (2 per cent), and allow to stand for a few minutes. If the solution becomes turbid, glue is present and should be determined as described in Section 24.

NOTE.—In the analysis of samples of hard rubber, disregard all reference to the Rubber Solvent Method.

## A. COMPLETE PROCEDURE

### Scope

8. (a) The methods given in the complete procedure cover the analysis of all types of rubber compounds to determine whether they conform to the chemical requirements of the specifications which are intended to secure compounds containing specified percentages of the best Hevea rubber and mineral fillers.

(b) Special methods are given for the analysis of compounds that contain glue, carbon, antimony, and waxy hydrocarbons.

(c) For the determination of the amount of rubber present in a vulcanized compound, the usual indirect method is given. This will give satisfactory results in all cases known today, except where there are found to be present decomposable compounding ingredients, such as carbonates that decompose at 550 C., cellulose, clay, asbestos, asbestine, talc, factice, and high percentages of mineral rubber. As practically all insulating compounds contain some of these ingredients, the said compounds, when specified, shall be analyzed in accordance with the Joint Rubber Insulation Committee's Procedure as described in Section 28.

(d) When carbonates that decompose at 550 C. are present, most accurate results are obtained by the use of the Joint Rubber Insulation Committee's Procedure as described in Section 28, or by the Rubber Solvent Method (Note) described in Sections 35 to 39, inclusive. When clay, asbestos, asbestine, or talc are present, none of the methods described herein is accurate unless correction is made for the loss of water of hydration on ashing clay, asbestos, asbestine, or talc. This correction can be made only if the nature and quantity of the fillers are known.

(e) If factice or high percentages of mineral rubber are used, no accurate method is known. If cellulose is present, the best results are obtained by the method described in Section 29.

(f) For determining the rubber hydrocarbon in hard rubber, best results may be obtained by using the Joint Rubber Insulation Committee's Procedure described in Section 28.

(g) A method employing a solvent for the vulcanized rubber has been included since it has proved to be more accurate than an ash determination when carbonates are present in a rubber compound. It may also be used in the analysis of asbestos rubber packings and rubberized fabrics.

NOTE.—In the analysis of samples of hard rubber, disregard all reference to the Rubber Solvent Method.

### Specific Gravity

9. (a) The specific gravity shall be determined by use of a pycnometer or by the method of hydrostatic weighings. Measurements shall be made at a temperature between 24.5 and 25.5 C. unless the coefficient of expansion of the rubber compound is known, in which case the determination shall be made at any convenient temperature and corrected to 25 C.

(b) *Pycnometer Method.*—Determine the specific gravity using the pycnometer with alcohol in place of water to eliminate the errors due to air bubbles.

$$\text{Sp. gr. (25/4 C.)} = 0.9971 \times \frac{W_1}{W_1 - (W_2 - W_3)} \times \text{sp. gr. of alcohol (25/25 C.)}$$

where:

$W_1$  = weight of specimen,

$W_2$  = weight of pycnometer filled with specimen and alcohol, and

$W_3$  = weight of pycnometer filled with alcohol.

(c) *Hydrostatic Method.*—This method provides for the determination of specific gravity through the loss in weight of a specimen when suspended in water. Dipping of the specimen in alcohol followed by blotting before suspending in water for weighing will aid in the elimination of bubbles which cause errors in the determination. A very fine wire is recommended as a supporting medium.

$$\text{Sp. gr. (25/4 C.)} = 0.9971 \times \frac{W_1}{W_1 - (W_2 - W_3)}$$

where:

$W_1$  = weight of specimen,

$W_2$  = weight of specimen and supporting wire in water, and

$W_3$  = weight of supporting wire in water.

### Total Extract

10. (a) *Extraction Apparatus.*<sup>4</sup>—The extraction apparatus used in this determination and for other extractions shall be of the general type and of the dimensions shown in either Fig. 1 or 2. The condenser and cover may be made of glass or of block tin.

(b) *Preliminary Extraction of Filter Paper.*—The ashless paper in which the sample is wrapped for this extraction shall be subjected to preliminary 2-hr. extractions with acetone and chloroform or a 2-hr. extraction with a mixture consisting of 32 per cent acetone and 68 per cent chloroform by volume.

(c) *Procedure.*—Place a weighed sample of approximately 2 g. in an extracted filter paper. If the sample is in the form of a sheet, cut it with scissors into strips 3 to 5 mm. in width. If the sample may become tacky during the extraction, take care that adjacent portions

<sup>4</sup> It is planned ultimately to replace the large type apparatus as shown in Fig. 1 by the smaller apparatus shown in Fig. 2. This smaller, lighter weight apparatus lends itself to more accuracy because of ease in cleaning and because of its light weight.

are separated by paper. Fold the paper so that it will fit in the extraction cup, and suspend the cup in a weighed extraction flask containing 50 to 75 ml. of a mixture consisting of 32 parts of acetone and 68 parts of chloroform by volume. (Prior to the weighing of the extraction flask, it shall have been dried for 2 hr. at 70 C. and cooled in a desiccator to the temperature of the balance.) Extract the sample continuously for 16 hr., heating at a rate such that the time required to fill and

chloroform extraction as described in Section 12 and add the value for chloroform extract to the result obtained for total extract. Distill off the solvent on a steam bath. Avoid allowing the flasks to stand on the steam bath after the solvent has been removed, because appreciable quantities of free sulfur may be lost in so doing. Dry the extraction flask and contents in an air bath for 2

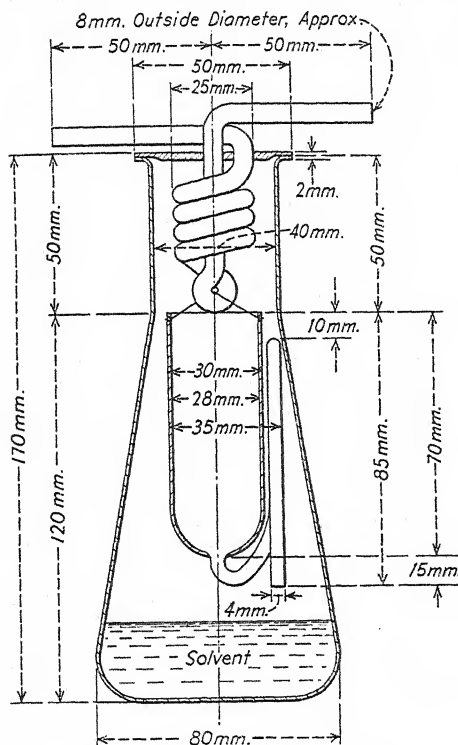


FIG. 1.—Extraction Apparatus.

empty the siphon cup will be between 2.5 and 3.5 min. (Hard rubber, and any soft rubber sample having a ratio of total sulfur to rubber hydrocarbon in excess of 10 per cent, shall be extracted for 72 hr.) Carefully note all characteristics of the extract, both when hot and cold. If the color is black, make a

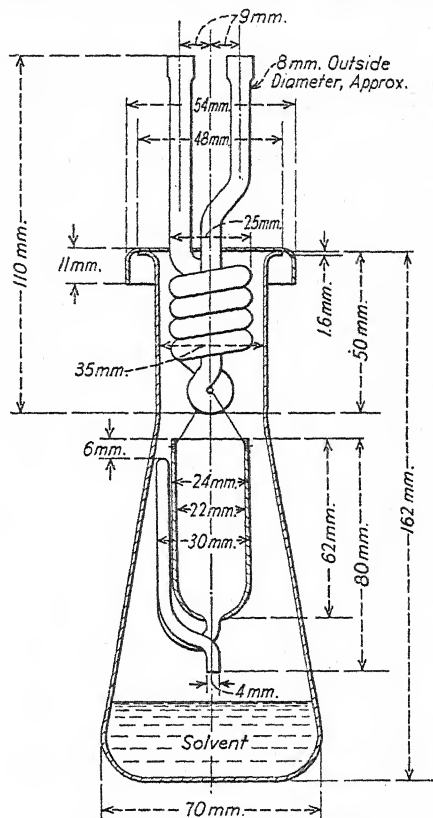


FIG. 2.—Extraction Apparatus.

hr. at 70 C. Cool in a desiccator to the temperature of the balance, and weigh.

$$\text{Total extract, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

#### Acetone Extract

11. (a) *Extraction Apparatus.*—The extraction apparatus used in this determination shall be that described in Section 10 (a).

(b) *Procedure*.—Place a weighed sample of approximately 2 g. in an extracted filter paper. If the sample is in the form of a sheet, cut it with scissors into strips 3 to 5 mm. in width. If the sample may become tacky during the extraction, take care that adjacent portions are separated by paper. Fold the paper so that it will fit in the extraction cup and suspend the cup in a weighed extraction flask containing 50 to 75 ml. of acetone. (Prior to the weighing of the extraction flask, it shall have been dried for 2 hr. at 70 C. and cooled in a desiccator to the temperature of the balance.) Extract the sample continuously for 16 hr., heating at a rate such that the time required to fill and empty the siphon cup will be between 2.5 and 3.5 min. (Hard rubber, and any soft rubber sample having a ratio of total sulfur to rubber hydrocarbon in excess of 10 per cent, shall be extracted for 72 hr.) Carefully note all characteristics of the extract, both when hot and cold. Distill off the acetone on a steam bath. Avoid allowing the flasks to stand on the steam bath after the solvent has been removed, because appreciable quantities of free sulfur may be lost in so doing. Dry the extraction flask and contents in an air bath for 2 hr. at 70 C. Cool in a desiccator to the temperature of the balance, and weigh.

$$\text{Acetone extract, uncorrected, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

#### Chloroform Extract

This determination is not applicable to crude, unvulcanized, or reclaimed rubber.

12. Suspend the extraction cup containing the rubber sample (Section 10 (c)) in a second weighed extraction flask containing 50 to 75 ml. of chloroform, and extract it for 4 hr. with chloroform, using the extraction rate prescribed in Section 10 (c). (Hard

rubber, and any soft rubber sample having a ratio of total sulfur to rubber hydrocarbon in excess of 10 per cent, shall be extracted for 24 hr.) Record the color of the chloroform solution. Evaporate the chloroform on a steam bath, taking care to avoid allowing the flasks to stand on the steam bath after the solvent has been removed. Dry the extraction flask and contents in an air bath for 2 hr. at 70 C. Cool in a desiccator to the temperature of the balance, and weigh. Reserve the extracted sample for extraction with alcoholic potash.

$$\text{Chloroform extract, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

#### Alcoholic-Potash Extract

13. Dry the rubber from the chloroform extraction (Section 12) at about 70 C. to remove the chloroform, transfer to a 200-ml. Erlenmeyer flask, add 50 ml. of alcoholic-potash solution, and heat under a reflux condenser for 4 hr. In the case of hard rubber, continue the heating for 16 hr. or more. Filter into a 250-ml. beaker, wash with two 25-ml. portions of boiling alcohol; then with three 25-ml. portions of boiling water, and evaporate the filtrate just to dryness. Use about 75 ml. of distilled water to transfer the residue to a separatory funnel: Acidify the solution with HCl (10 per cent), testing with Congo red paper. Extract with four 25-ml. portions of ether, unless the fourth portion should be colored, when the extraction shall be continued until no further quantity can be removed. Unite the ether fractions and wash thoroughly with distilled water until free from acid (two washings are generally sufficient). Filter the ether solution through a plug of absorbent cotton into a weighed flask, wash with ether, evaporate, dry to

constant weight at 70 C., cool, and weigh.

$$\text{Alcoholic-potash extract, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

#### Total Sulfur, Recommended Method

14. (a) When this method is used the sulfur determined represents the sulfur exclusive of that originally present in barytes or combined during analysis with barium salts if these are present in the compound.

(b) Place 0.5 g. of soft rubber or 0.2 g. of hard rubber in a 500-ml. Erlenmeyer destruction flask of chemically resistant material.<sup>5</sup> Add 10 ml. of zinc oxide-nitric acid solution and moisten the sample thoroughly. Let stand at least 1 hr., overnight if convenient. By so doing the sample becomes partly decomposed; this permits the addition of fuming nitric acid with no danger of ignition of the sample. Add 15 ml. of fuming HNO<sub>3</sub> and whirl the flask rapidly to keep the sample immersed to avoid ignition. With some samples it may be necessary to cool the flask under running water.

(c) When the solution of the rubber appears to be complete, add 5 ml. of a saturated water solution of bromine and slowly evaporate the mixture to a foamy syrup. (For the determination of total sulfur in unvulcanized mixtures, use 3 ml. of bromine in place of bromine water.)

(d) If organic matter or carbon remain at this point, add a few milliliters of fuming HNO<sub>3</sub> and a few crystals of potassium chlorate (*Caution*) and evaporate at a boil. Repeat this operation until all carbon is gone and the solution is clear, colorless or light yellow.

(e) At this point either of the following procedures may be used:

(1) Place the flask on an asbestos

gauze and evaporate the mixture to dryness over a Tirrill burner. Then bake the mixture at the highest temperature of the burner until all nitrates are decomposed and no more nitrogen oxide fumes can be detected. The flask must be carefully annealed after this procedure by gradually decreasing the flame or by placing the flask on successively cooler sources of heat.

(2) Evaporate the mixture, cool, add 10 ml. of HCl (sp. gr. 1.19), and evaporate to dryness, avoiding spattering. Repeat this procedure once, or more than once if oxides of nitrogen are still evolved.

(f) Cool the flask, add 50 ml. of HCl (1:6) and digest warm until solution is as complete as possible. In case the original mixture contains barium sulfate or other barium salts, they will be precipitated at this point with consequent loss of sulfur in the case of the latter. If litharge is present in moderate quantities, it will not interfere under the existing conditions if all filtrations and washings are made hot. Filter the solution, wash the filter, and dilute the solution to 300 ml. Add 10 ml. of saturated picric acid solution, precipitate with BaCl<sub>2</sub> (100 g. per l.), and allow the precipitate to stand overnight. Filter and wash with hot water until the filter is colorless, and determine the barium sulfate precipitate in the usual manner.

$$\text{Total sulfur, per cent} = \frac{\text{wt. of BaSO}_4 \times 0.1373}{\text{wt. of sample}} \times 100$$

#### Total Sulfur, Alternate Method

15. (a) When this alternate method is used, the sulfur determined is all of the sulfur originally present including that in barytes. In order to obtain the total sulfur as defined in Section 2 (e), correction must be made by subtracting

<sup>5</sup> Pyrex, quartz glass, or similarly resistant material is satisfactory.

the sulfur originally present in the barytes from the result of this determination.

(b) Place 0.5 g. of soft rubber or 0.2 g. of hard rubber in a porcelain crucible of about 75-ml. capacity, add 15 ml. of the nitric acid-bromine mixture, cover the crucible with a watch glass, and let it stand for 1 hr. in the cold. Heat for 1 hr. on the steam bath, remove the cover, rinse it with a little distilled water, and evaporate to dryness. Add 3 ml. of  $\text{HNO}_3$  (sp. gr. 1.42), cover, warm a short time on the steam bath, then let it cool. Carefully add in small portions, by means of a glass spatula, 5 g. of sodium carbonate (weighed to 0.5 g.). Raise the watch glass only high enough to permit the introduction of the spatula. Allow the carbonate to slide down the side of the crucible as it must not be dropped directly into the acid. Rinse the watch glass with 2 or 3 ml. of hot distilled water and stir the mixture thoroughly with a glass rod. Digest for a few minutes, spread the mixture halfway up the side of the crucible to facilitate drying, and dry on a steam bath. Fuse the mixture by heating over a sulfur-free flame.

(c) Place the crucible in an inclined position on a wire triangle and start the ignition over a low flame. The tendency for the organic matter to burn too briskly may be controlled by judicious use of the stirring rod with which the burning portion is scraped away from the rest. When part of the mass is burned white, work a fresh portion into it until all of the organic matter is destroyed. It is necessary to hold the edge of the crucible with tongs. Toward the last half of the operation the flame should be increased. It is unnecessary to heat the crucible to redness. With care, a crucible can be used for at least 10 to 12 fusions.

(d) After a fusion, allow the crucible to cool, place it in a 400-ml. beaker, add

sufficient distilled water to cover the crucible (about 125 ml.), and digest on the steam bath or plate overnight.

(e) Filter the solution into a covered 400-ml. beaker containing 5 ml. of  $\text{HCl}$  (sp. gr. 1.19), and wash the residue thoroughly with hot sodium carbonate solution (5 per cent). A qualitative test for barium should be made on the residue to ascertain whether it is necessary to determine barium in the ash according to Section 20. Complete the acidification of the filtrate and washings and add 2 ml. of  $\text{HCl}$  (sp. gr. 1.19) in excess. Cover the beaker and heat the solution on a steam bath. The total volume of the solution should be 300 ml. The solution shall be acid to Congo paper in order to insure the complete destruction of the carbonates. Add 10 ml. of a saturated aqueous solution of picric acid to assist in barium sulfate crystal growth and to serve as an index of thorough washing. Precipitate with barium chloride and determine the sulfur in the usual manner.

$$\text{Sulfur, per cent} = \frac{\text{wt. of BaSO}_4 \times 0.1373}{\text{wt. of sample}} \times 100$$

$$\text{Total sulfur, per cent} = \text{percentage sulfur determined} - \text{percentage sulfur in barium sulfate}$$

### Free Sulfur

16. (a) *Recommended Method*.—Add to the flask containing the acetone extract, 10 ml. of zinc oxide-nitric acid mixture and 2 to 3 ml. of bromine and cover with a watch glass. Allow to stand near the steam plate for 30 min., then heat on a steam plate to a foamy syrup. Add 10 ml. of fuming  $\text{HNO}_3$ , heat on the hot plate with the cover removed until all bromine is expelled. Continue the determination as described in Section 14 (d), (e), and (f).

(b) *Alternate Method*.<sup>5a</sup>—Place 2 g. of a sample thinly sheeted, 0.05 to 0.075

<sup>5a</sup> This alternate method for the determination of free sulfur is not applicable to hard rubber.

cm. (0.02 to 0.03 in.) in a 400-ml., thin-walled, chemically resistant glass flask.<sup>5</sup> Add 100 ml. of sodium sulfite solution (5 per cent), 5 ml. of a sodium stearate suspension in water (0.1 per cent), and approximately 1 g. of paraffin. Cover the flask with a small watch glass and heat so as to boil gently for 2 hr. Remove the flask and add 100 ml. of strontium chloride (0.5 per cent) and 10 ml. of cadmium acetate (3 per cent). Separate the rubber and precipitates by filtration, using a Büchner funnel with suction. (The funnels are prepared by forming a thin asbestos pad over a single sheet of qualitative filter paper. Filters thus prepared can be used numerous times.) Wash with two 75 to 100-ml. portions of a wash solution containing 40 ml. of cadmium acetate (3 per cent) per liter. To the filtrate, add while stirring 5 ml. of 40 per cent formalin solution, 10 ml. of glacial acetic acid, and 5 ml. of starch solution (1 per cent). Add enough crushed ice to bring the temperature of the solution below 15 C., and titrate with a standard solution of iodine to a blue end point.

Run a blank determination on the reagents, and subtract this figure, usually 0.2 to 0.3 ml., from the titrations on the samples.

#### Ash, Rapid Method

17. (a) Wrap a 1-g. sample in filter paper, extract with acetone for 4 hr., and transfer to a weighed, approximately 50-ml. porcelain crucible. For hard rubber the extraction may be eliminated. Use a 2-g. sample for the analysis of hard rubber. Ash the sample in a muffle furnace by heating at the following rate:

Time, min. . . .	0	5	10	15	70	75	80	85	145
Temperature, deg. Cent. . . .	0	100	200	300	300	400	500	550	550

(b) Remove the crucible from the furnace, cool, and weigh.

$$\text{Ash, per cent} = \frac{\text{wt. of ash}}{\text{wt. of sample}} \times 100$$

(c) If no furnace is available, distill off the rubber over a very small flame, not allowing it to catch fire, and ignite gently until burnt clean. Cool, weigh, and determine ash in the usual manner.

#### Ash, Referee Method

18. (a) *Apparatus*.—In case of disagreement on results of determining the percentage of ash by the rapid method described in Section 17, an electric muffle furnace shall be employed for the test. Numbered porcelain crucibles 41 mm. in diameter and 25 mm. in height shall be used. The bottom of the furnace shall be covered by a sheet of asbestos 0.06 in. in thickness, cut to fit.

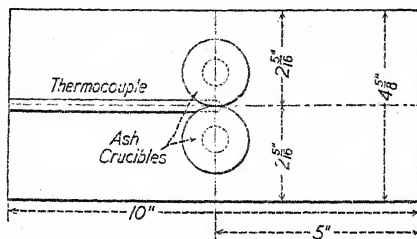


FIG. 3.—Location of Apparatus for Ash Determination (Referee Method).

(b) *Procedure*.—Extract a 2-g. sample with acetone as described in the acetone extraction (see Section 11). Allow the extracted sample to dry at 100 C., cool in a desiccator, and weigh. Divide the sample into two parts of equal weight, place each of them in crucibles previously ignited, cool in a desiccator, and weigh. Place the two crucibles, with contents, together (with their tops touching) in the furnace on a line crossing the furnace half-way between its ends, as shown in Fig. 3. Regulate the temperature of the furnace by means of a rheostat so that the temperature corresponds to the curve shown in Fig. 4, with a maximum permissible variation



of plus or minus 25 C. Measure the temperature with a thermocouple (previously calibrated) enclosed in a quartz tube closed at one end, 6 to 7 mm. in diameter, 0.5 to 1 mm. in thickness, and placed in a horizontal position in contact with the asbestos sheet on the bottom of the furnace, so that the hot junction of the couple is on a line crossing the furnace half-way between its ends, and half-way between the two crucibles, as shown in Fig. 3. Adjust the door of the furnace to conform to the following requirements:

	Opening
First 400 min.....	$\frac{1}{2}$ in.
From 400 to 450 min.....	$1\frac{1}{2}$ in.
Remainder of period (7 min.).....	wide open

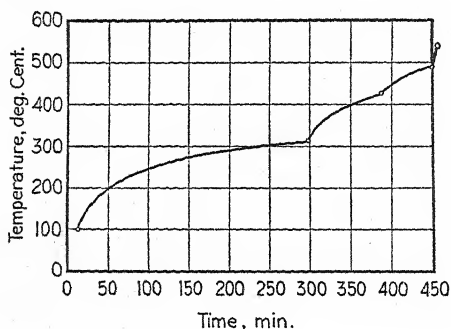


FIG. 4.—Time-Temperature Curve for Ash Determination (Referee Method).

(c) At the end of the period of heating, as shown, remove the crucibles from the furnace, cool in a desiccator, and weigh.

$$\text{Ash, per cent} = \frac{\text{wt. of ash}}{\text{wt. of sample}} \times 100$$

#### Sulfur in Ash

19. (a) Add 3 ml. of nitric acid-bromine mixture to the ash (see Section 17), cover with a watch glass and heat for 1 hr., remove the cover, rinse it with a little distilled water, and evaporate to dryness. Complete the determination of sulfur as described in Section 15.

(b) Save the insoluble residue, after filtering the solution of the fusion mix-

ture in water, for testing in accordance with Section 20.

#### Sulfur as Barium Sulfate

20. Calculate the sulfur as barium sulfate from the barium in the ash, which shall be determined as follows:

Wash the insoluble matter, referred to in Section 19 (b), back into the original beaker with hot water, dissolve the residue in the beaker and any traces on the filter paper with HCl (sp. gr. 1.19) and heat the solution on the steam bath. Filter and wash thoroughly with hot water. Adjust the acidity, by means of 6 N  $\text{NH}_4\text{OH}$  and 6 N HCl to be between 0.2 and 0.3 N in HCl. Cool, saturate the solution with  $\text{H}_2\text{S}$ , and when the lead sulfide has settled, filter into a 400-ml. beaker and wash thoroughly. The total volume of the solution shall not be over 200 ml. Precipitate the barium with  $\text{H}_2\text{SO}_4$  (10 per cent) and treat the precipitated barium sulfate in the usual manner as for sulfur determination. Barium sulfate determined above is assumed to have been added as such, and the value will usually be less than the amount of commercial barium sulfate originally compounded. Conversely, because of sulfate and phosphate impurities often present in commercial barium sulfate, the value for total sulfur exclusive of that from barium sulfate will frequently be high when the calculation is made with data obtained from this procedure and that described in Section 15. Obviously, if barium carbonate is present, it must be determined in order that an undue correction will not be made.

Sulfur as barium sulfate, per cent =

$$\frac{\text{wt. of BaSO}_4 \times 0.1373}{\text{wt. of sample}} \times 100$$

#### Total Antimony

21. When a qualitative test indicates that antimony is present, weigh out a

0.5-g. sample, and transfer to a Kjeldahl flask, add 25 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) and 10 to 12 g. of potassium sulfate, place a funnel in the neck of the flask, and heat until the solution becomes colorless. Cool, and wash the funnel, dilute the solution to 100 ml. with water, and transfer to a 400-ml. beaker. Dilute to 250 ml. with hot water, and precipitate the antimony with  $\text{H}_2\text{S}$ . Filter, and transfer the precipitate to a Kjeldahl flask, add 15 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84), 10 to 12 g. of potassium sulfate, and heat as described above until the solution is colorless. Wash the funnel, dilute the solution to 100 ml. with water, add 1 to 2 g. of sodium sulfite, and boil until all the sulfur dioxide is driven out. This is shown when no blue color is obtained with starch iodate paper. Add 25 ml. of  $\text{HCl}$  (sp. gr. 1.19), dilute to 200 ml., regulate the temperature to about 60 C., add 2 drops of 0.2 per cent methyl red solution, and titrate with standard potassium bromate until the solution is colorless. When the indicator starts to fade, add the bromate slowly, using another drop of indicator if desired. At the end point, an added drop of indicator should become colorless. If iron is found to be absent it is not necessary to precipitate the antimony with  $\text{H}_2\text{S}$  and the second heating in the Kjeldahl flask may be eliminated.

$$\text{Antimony, per cent} = \frac{\text{Sb equivalent of KBrO}_3 \times \text{ml. of KBrO}_3}{\text{wt. of sample}} \times 100$$

#### Antimony in the Ash

22. Antimony in the ash shall be determined on the ash of a 1-g. sample as follows: Transfer the ash to a 600-ml. Erlenmeyer flask, add 12 to 15 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) and 10 to 12 g. of potassium sulfate, and boil until solution is complete. It may be necessary

to warm part of the  $\text{H}_2\text{SO}_4$  in the crucible to transfer any adhering particles to the flask. Rinse with the remaining portion of acid. Then complete the determination as described in Section 21.

$$\text{Antimony, as percentage Sb}_2\text{O}_3 \text{ in ash} = \frac{\text{Sb equivalent of KBrO}_3 \times \text{ml. of KBrO}_3 \times 1.26}{\text{wt. of sample}} \times 100$$

#### Free Carbon

23. (a) Extract a 0.5-g. sample for 8 hr. with a mixture of 68 per cent chloroform and 32 per cent acetone by volume.

(b) Transfer the sample to a 250-ml. beaker and heat on the steam bath until it no longer smells of chloroform. Add a few milliliters of  $\text{HNO}_3$  (sp. gr. 1.42) and allow to stand for about 10 min. Add 50 ml. more of  $\text{HNO}_3$  (sp. gr. 1.42), taking care to wash down the sides of the beaker, and heat on the steam bath for at least 1 hr. At the end of this time there should be no more bubbles or foam on the surface. Pour the liquid while hot into a Gooch crucible, taking care to keep as much as possible of the insoluble material in the beaker. Filter slowly with gentle suction and wash well by decantation with hot  $\text{HNO}_3$  (sp. gr. 1.42). (*Caution:* Empty the filter flask.) Wash with acetone and a mixture of equal parts of acetone and chloroform until the filtrate is colorless. Digest the insoluble material, which has been carefully retained in the beaker, for 30 min. on the steam bath with 35 ml. of  $\text{NaOH}$  solution (25 per cent). This treatment with alkali may be omitted if silicates are absent. Dilute to 60 ml. with hot distilled water and heat on the steam bath. Filter the solution of alkali and wash well with hot  $\text{NaOH}$  solution (15 per cent).

(c) Next, wash the residue about four times with hot  $\text{HCl}$  (sp. gr. 1.19). Neutralize the last washing with  $\text{NH}_4\text{OH}$

and test for the presence of lead with sodium chromate solution. If lead is present continue to wash with hot HCl (sp. gr. 1.19) and, finally, wash with warm HCl (5 per cent). Remove the crucible from the funnel, taking care that the outside is perfectly clean, dry it in an air bath for  $1\frac{1}{2}$  hr. at 110 C., cool, weigh, burn off the carbon at a dull red heat, and reweigh. The difference in weight represents approximately 105 per cent of the carbon originally present in the form of lampblack or gas black.

$$\text{Free carbon, per cent} = \frac{a - b}{1.05 \times \text{wt. of sample}} \times 100$$

where:

$a$  = weight of crucible with carbon, and

$b$  = weight of crucible after ignition.

#### Nitrogen Calculated as Glue

24. (a) When glue is found to be present, a correction shall be made for the natural protein in the rubber. The average amount of nitrogen is 0.4 per cent. See Section 32 (b) for calculations.

(b) Extract a 2-g. sample for 8 hr. with acetone. Remove the solvent from the sample, and transfer the latter from the filter paper to a 750-ml. Kjeldahl flask. Add 25 to 30 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84), 10 to 12 g. of sodium sulfate, and about 1 g. of copper sulfate. Heat gently until the first vigorous frothing ceases, then raise the heat gradually until the liquid boils. Continue the boiling until the solution becomes clear. Allow the flask to cool, dilute carefully with 150 ml. of water, and again allow to cool. Add 100 ml. of sodium hydroxide solution (50 per cent), pouring it carefully down the side of the flask, so that it does not mix immediately with the acid solution. Add about 1 g. of granulated zinc to prevent bumping and

a piece of paraffin the size of a pea to diminish frothing. Connect the flask quickly with a condenser, the delivery tube of which dips into a 500-ml. Erlenmeyer flask, containing 50 ml. of  $\text{H}_2\text{SO}_4$  (0.1  $N$ ) diluted to about 100 ml. Carefully swirl the flask to mix the contents and start to heat gently, increase the flame as the danger of foaming over diminishes, and finally boil briskly until about one-half of the liquid has passed over into the receiver. Add methyl red solution and titrate the excess acid by means of NaOH (0.1  $N$ ). A blank determination shall be made.

$$\text{Nitrogen as glue, per cent} = \frac{100 (\text{H}_2\text{SO}_4 \times \text{normality} - \text{NaOH} \times \text{normality}) (0.014) (6.5)}{\text{wt. of sample}}$$

When a 2-g. sample is used this formula becomes:

$$\text{Nitrogen as glue, per cent} = (\text{H}_2\text{SO}_4 \times \text{normality} - \text{NaOH} \times \text{normality}) \times 4.55$$

#### Unsaponifiable Matter

25. (a) If this determination is made, the free sulfur shall be determined on a separate 2-g. sample.

(b) Add to the acetone extract obtained from two 2-g. samples (see Section 11 (b)), 50 ml. of a normal alcoholic-potash solution, heat on the steam bath under a reflux condenser for 2 hr., remove the condenser and evaporate to dryness. Transfer to a separatory funnel, using about 100 ml. of water, add 25 ml. of ether, and shake. Allow the two layers to separate thoroughly, then draw off the water layer. Continue the extraction of the water layer with fresh portions of ether until no more unsaponifiable matter is removed, unite the ether layers, and wash with distilled water until a negative test with phenolphthalein is obtained on the wash water. Transfer the ether to a weighed flask, distill off the ether, dry to constant weight at 70 C., cool, and weigh.

$$\text{Unsaponifiable matter, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

### Hydrocarbons A

26. (a) To the unsaponifiable matter (see Section 25), add 50 ml. of absolute alcohol, and heat on the steam bath for 30 min. Let the flask stand in a mixture of ice and salt for 1 hr. Filter off the separated waxy hydrocarbons on filter paper, by applying a gentle suction. Wash with alcohol (95 per cent) which has been cooled in an ice-salt mixture. The funnel should be surrounded by a freezing mixture.

(b) Dissolve the precipitate from the filter paper with hot chloroform, and catch the solution in a weighed 100 to 150-ml. beaker. Wash the flask with hot chloroform, which is added to the same beaker in order to include any insoluble matter adhering to the walls of the flask. Evaporate the solvent, dry to constant weight at 70 C., cool, and weigh.

$$\text{Hydrocarbons A, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

### Hydrocarbons B

27. Evaporate the alcohol from the flask containing the alcohol-soluble unsaponifiable material, add 25 ml. of carbon tetrachloride, and transfer to a separatory funnel. Shake with  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84), drain off the discolored acid, and repeat with fresh portions of acid until there is no longer any discoloration. After drawing off all the acid, wash the carbon tetrachloride solution with repeated portions of water until all traces of acid are removed. Transfer the carbon tetrachloride solution to a weighed flask, evaporate the solvent, and dry to constant weight at 70 C., cool, and weigh.

$$\text{Hydrocarbons B, per cent} = \frac{\text{wt. of extract}}{\text{wt. of sample}} \times 100$$

### Rubber Hydrocarbons

28. *Joint Rubber Insulation Committee's Procedure.*—Add to the flask containing the rubber residue from the alcoholic-potash extraction sufficient water to make the total 125 ml., and then add 25 ml. of HCl (sp. gr. 1.19). Heat for 1 hr. at 100 C. Decant the supernatant liquid through a hardened filter paper on a Büchner funnel, 7 cm. in diameter, using suction. Wash the residue with 25 ml. of hot water and decant. (Although a Büchner funnel is recommended, it is permissible to use an 11-cm. hardened filter paper with platinum cone in a 60-deg. funnel.) Perform this entire treatment with water and HCl (sp. gr. 1.19) three times. The rubber at this stage should be white and practically free from black specks of undissolved fillers; if not, continue the acid treatment until the black specks disappear. (If carbon is present, all the particles of rubber will be grayish, bluish, or black, depending on the form and quantity of carbon used. Black specks in light particles of rubber usually indicate the presence of lead sulfide which must be removed to prevent the formation of lead sulfate on igniting the residue C.) Add 150 ml. of hot water to the flask and allow to stand on a steam bath or hot plate for 30 min. and decant through the filter paper, repeating until washings are free from chlorides. Transfer all the rubber in the flask to the filter paper and dry as much as possible by suction. Wash the rubber with 50 ml. of alcohol (95 per cent), using suction. Transfer the entire residue to a weighing bottle. Dry at 95 to 100 C. for 1 hr., cool in a vacuum desiccator under reduced pressure, and weigh. Dry for 30 min., cool, and weigh, repeating this process until either constant weight is reached or the weight starts to increase. Let this weight be represented by C. The weight originally taken for the ace-

tone extract determination shall be called *A*. Determine the ash, *E*, (Note) on a portion, *D*, of this residue, *C*, and the sulfur, *H*, on the remaining portion, *G*. Also determine the sulfur, *F*, on the ash, *E*. Perform the ash determination as described in Section 17, and the sulfur determination as described in Section 15.

$$\text{Rubber hydrocarbons, per cent} = 100 \frac{C}{A} \left( 1 - \frac{H}{G} - \frac{E-F}{D} \right)$$

NOTE.—See the statement regarding decomposable fillers, Section 8 (*d*).

### Cellulose

29. (*a*) Treat 0.5 g. of rubber previously extracted with a constant-boiling mixture of acetone and chloroform with 25 ml. of rubber-solvent oil (see Section 3 (*i*)), in a 250-ml. lipped assay flask. Place in an air bath at a temperature of 145 to 150 C. until small particles can be seen distinctly in the supernatant liquid, which requires about 3 hr. Dilute with 15 ml. of benzene and 200 ml. of petroleum ether. Filter through a Gooch crucible and wash the residue thoroughly with warm acetone. Treat repeatedly with hot HCl (10 per cent) until the residue is free from acid-soluble fillers. Wash the pad well with boiling water, and run small portions of acetone through it until the filtrate is colorless. Wash with alcohol and dry at 105 C. to constant weight. Remove the pad from the crucible with the help of a pair of sharp-pointed tweezers, using the under portion of the pad as a swab to clean the sides of the crucible, and place all of this material in a tared weighing bottle. Replace in the drying oven for about 10 min., cool, and weigh. Weight of weighing bottle, pad, insoluble fillers, and cellulose minus weight of the weighing bottle equals weight of pad, insoluble fillers, and cellulose.

(*b*) Transfer the contents of the weighing bottle to a 50-ml. beaker and pour

over it 15 ml. of acetic anhydride and 0.5 ml. of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84), and allow to digest for at least 1 hr., on the steam bath. After the mixture has cooled thoroughly, dilute with 25 ml. of acetic acid (90 per cent), and filter through a weighed Gooch crucible. To guard against traces of the material being carried through, this filtration, as well as the ones to follow, shall be very slow and only gentle suction shall be used. Wash with hot acetic acid (90 per cent), until the filtrate comes through absolutely colorless and then wash about four times more. Wash with acetone about five times. After having taken care that all of the material has been washed out of the beaker in which the acetylation took place, remove the crucible from the funnel, clean the outside thoroughly, and dry for 2 hr. at 150 C. Cool, and weigh. The original weight of the crucible plus weight of pad, fillers, and cellulose minus the weight of the crucible after acetylation equals cellulose.

$$\text{Cellulose, per cent} = \frac{\text{cellulose}}{\text{wt. of sample}} \times 100$$

### Barium Carbonate

30. (*a*) Barytes was determined (Section 20) by the calculation to barytes of all barium found in the sample. Obviously, if barium carbonate is present, it must be determined in order that an undue correction will not be made. The determination shall be made as follows: Place a 1-g. sample, in a porcelain boat, and place this in a combustion tube through which passes a current of carbon dioxide. Ash the sample in the tube. After ignition and cooling, in the atmosphere of carbon dioxide, remove the boat, finely grind the residue in an agate mortar, transfer it to a 250-ml. beaker, and treat with 5 to 10 g. of ammonium carbonate, 15 to 20 ml. of strong ammonia water, and about 50 ml. of distilled water. Boil the mixture for

20 min., filter, and wash the precipitate thoroughly to remove all soluble sulfates. Wash the residue on the filter paper back into the original beaker and add about 10 ml. of glacial acetic acid with sufficient water to make the total volume about 100 ml. Heat this to boiling and filter through the same paper as before. Pass hydrogen sulfide into the filtrate to precipitate the lead and treat the solution subsequently as in the determination of barytes. The final weight of barium sulfate obtained shall be calculated to barium carbonate.

$$S = \frac{\text{wt. of BaSO}_4 \times 0.1373}{\text{wt. of sample}} \times 100$$

where:

$S$  = the percentage of sulfur required to convert barium present as carbonate to sulfate.

(b) The Rubber Solvent Method (Note) described in Sections 35 to 39, inclusive, has been found to be more rapid in the determination of barium carbonate, and as accurate as the above method.

NOTE.—In the analysis of samples of hard rubber, disregard all reference to the Rubber Solvent Method.

#### Analysis of Ash

31. Analyze one sample of ash (Section 17) for silicon dioxide and insoluble matter, lead, iron and aluminum oxides, calcium oxide, and magnesium oxide as described in the following Paragraphs (a) to (e). Analyze another ash sample for zinc oxide as described in Paragraph (f). Use 1-g. samples for soft rubber products and 2-g. samples for hard rubber products.

(a) *Silicon Dioxide and Insoluble Matter*.—Dissolve one of the samples of ash in 10 ml. of HCl (sp. gr. 1.19), rinse the crucible thoroughly, dilute to 100 ml., and evaporate to dryness in a casserole.

Bake for 1 hr. at 110 C. Moisten with 10 ml. of HCl (sp. gr. 1.19) and 3 drops of HNO<sub>3</sub> (sp. gr. 1.42), and digest for 15 min. on the steam bath. Add 100 ml. of water, boil, filter, and wash with hot water. Dry and ignite in a porcelain crucible. Weigh to determine the SiO<sub>2</sub> and insoluble matter. If the residue is large enough to justify an analysis for SiO<sub>2</sub>, transfer to a platinum crucible and add 2 to 3 ml. of HF and a few drops of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84). Evaporate to dryness, and carefully ignite at a low red heat. The loss in weight is SiO<sub>2</sub>. Calculate the percentages of SiO<sub>2</sub> and of SiO<sub>2</sub> and insoluble matter as follows:

SiO<sub>2</sub> and insoluble matter, per cent =

$$\frac{A - B}{C} \times 100$$

$$\text{SiO}_2, \text{ per cent} = \frac{(A - B) - (D - E)}{C} \times 100$$

where:

$A$  = weight of residue and porcelain crucible after ignition,

$B$  = weight of porcelain crucible,

$C$  = weight of original sample,

$D$  = weight of residue and platinum crucible after treatment with HF and ignition, and

$E$  = weight of platinum crucible.

A large residue after the HF treatment may be BaSO<sub>4</sub>, PbSO<sub>4</sub>, TiO<sub>2</sub>, which may be identified by microscopic examination. If small amounts of Al<sub>2</sub>O<sub>3</sub> and Fe<sub>2</sub>O<sub>3</sub> are present in the residue, they may be dissolved by fusion with potassium pyrosulfate, reprecipitated with NH<sub>4</sub>OH, and added to the R<sub>2</sub>O<sub>3</sub> precipitate, provided TiO<sub>2</sub> is absent and an exact analysis for Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, or both is desired.

(b) *Lead*.—A complete precipitation of the lead as PbS may be made if the concentration and acidity are carefully controlled. Just neutralize the filtrate

from the determination of silicon dioxide and insoluble matter with  $\text{NH}_4\text{OH}$ , and add 1 ml. of  $\text{HCl}$  (sp. gr. 1.19). Run a rapid stream of  $\text{H}_2\text{S}$  into the solution and dilute to between 50 and 100 ml. Continue the addition of  $\text{H}_2\text{S}$  until precipitation is complete. Filter, and wash with a saturated solution of  $\text{H}_2\text{S}$ . If antimony is present, it will precipitate under these conditions; zinc may also be precipitated, but neither will interfere with the determination of lead. Dissolve the  $\text{PbS}$  in diluted  $\text{HNO}_3$  (1:1); boil to complete solution. If antimony is present it may not be dissolved by this procedure. Filter. Cool the filtrate, add 10 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84), and evaporate to dense white fumes of  $\text{H}_2\text{SO}_4$ . Cool, dilute with 50 ml. of water, add an equal volume of 95 per cent alcohol, and let stand overnight. Filter on a tared Gooch crucible, wash with 50 per cent alcohol, and dry at 105 C. Calculate the percentage of lead as follows:

$$\text{Lead, per cent} = \frac{(A - B) \times 0.6832}{C} \times 100$$

where:

$A$  = weight of crucible and  $\text{PbSO}_4$ ,  
 $B$  = weight of crucible,  
 $C$  = weight of original sample,  
 and

0.6832 = conversion factor from  $\text{PbSO}_4$  to metallic lead.

(c) *Iron and Aluminum Oxides ( $\text{R}_2\text{O}_3$ ).*—Boil the filtrate from the lead sulfide vrecipitation to expel  $\text{H}_2\text{S}$ . Adjust the volume of solution to 100 to 150 ml. Add a few drops of  $\text{HNO}_3$  and boil the solution again. Test for ferrous iron, using  $\text{K}_3\text{Fe}(\text{CN})_6$  as an outside indicator on a spot plate. If ferrous iron is present, add more  $\text{HNO}_3$  and proceed as before until all the iron is oxidized. Add 5 g. of solid  $\text{NH}_4\text{Cl}$ . Add  $\text{NH}_4\text{OH}$  (sp.

gr. 0.90) until the solution is colored definitely yellow by methyl red, but do not add an excess. Heat to boiling and boil for 5 min. When the precipitate has settled, filter, with the aid of filter pulp if the precipitate is large, and wash with  $\text{NH}_4\text{Cl}$  (2 per cent). Carefully char off the filter paper at low temperature and ignite the residue in a freely oxidizing atmosphere. Calculate the percentage of  $\text{R}_2\text{O}_3$  as follows:

$$\text{R}_2\text{O}_3, \text{ per cent} = \frac{(A - B)}{C} \times 100$$

where:

$A$  = weight of crucible and  $\text{R}_2\text{O}_3$   
 ( $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$ ),

$B$  = weight of crucible, and

$C$  = weight of original sample.

Iron in the  $\text{R}_2\text{O}_3$  residue may be determined, if desired, by fusing the residue with potassium pyrosulfate, dissolving the melt in 6  $N$   $\text{H}_2\text{SO}_4$ , reducing the iron with amalgamated zinc, and titrating the iron with  $\text{KMnO}_4$ .

(d) *Calcium Oxide.*—Zinc must be removed before the determination of calcium and magnesium oxides. Dilute the filtrate from the  $\text{R}_2\text{O}_3$  determination to 250 ml. Make the solution just acid with 6  $N$   $\text{HCl}$ . Pass in a rapid stream of  $\text{H}_2\text{S}$ , allow the precipitate to settle, and filter. Test for complete removal of zinc with  $(\text{NH}_4)_2\text{S}$  in alkaline solution. Filter again if necessary. Make the filtrate just acid with 6  $N$   $\text{HCl}$ , and evaporate to 150 ml. Filter to remove sulfur. Add methyl red indicator, heat to 50 C., neutralize with  $\text{NH}_4\text{OH}$  (sp. gr. 0.90), and add 1 ml. in excess. Make the solution just acid with a solution of oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ) (10 per cent), add 12 ml. in excess, and boil for 2 min., while stirring vigorously. Add approximately 50 ml. of saturated ammonium oxalate ( $(\text{NH}_4)_2\text{C}_2\text{O}_4$ ) (4 per cent), adding more if the solution is still



acid to methyl red, dilute to 250 to 300 ml., boil for 2 min., and digest on a steam bath for 1 hr. Allow to cool, filter, and wash with a solution containing 0.2 per cent of  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  and 0.1 per cent of  $\text{H}_2\text{C}_2\text{O}_4$ . Dissolve the precipitate in 50 ml. of warm 3 *N* HCl and reprecipitate by the above procedure, uniting filtrate and washings with the first filtrate and washings. The quantities of  $\text{H}_2\text{C}_2\text{O}_4$  and  $(\text{NH}_4)_2\text{C}_2\text{O}_4$  used in the reprecipitation may be one-half or possibly only one-fourth as large as in the first precipitation. If CaO is to be determined volumetrically, wash the precipitate of  $\text{CaC}_2\text{O}_4$  finally with water, dissolve the precipitate from the paper in hot 6 *N*  $\text{H}_2\text{SO}_4$ , and titrate hot with standard 0.1 *N*  $\text{KMnO}_4$ ,<sup>6</sup> finally adding the filter paper to the mixture and finishing the titration rapidly. A sintered glass crucible of fine porosity may be advantageously substituted for the filter paper. If CaO is to be determined gravimetrically, dry and ignite the precipitate in a covered procelain crucible, the ignition temperature being from 1000 to 1200 C. Calculate the percentage of CaO as follows:

Volumetric method:

$$\text{CaO, per cent} = \frac{A \times N \times 0.028}{C} \times 100$$

where:

*A* = milliliters of  $\text{KMnO}_4$ ,  
*N* = normality of  $\text{KMnO}_4$ ,  
*C* = weight of original sample, and  
 0.028 = grams of CaO equivalent to each milliliter of exactly 1 *N*  $\text{KMnO}_4$ .

Gravimetric method:

$$\text{CaO, per cent} = \frac{A - D}{C} \times 100$$

where:

*A* = weight of precipitate plus crucible,  
*D* = weight of crucible, and  
*C* = weight of original sample.

(*e*) *Magnesium Oxide*.—Evaporate to dryness the combined filtrates and washings from the determination of calcium. Add 50 ml. of  $\text{HNO}_3$  (sp. gr. 1.42), cover, and warm until evolution of gas subsides. Uncover and evaporate to dryness, avoiding spattering. Heat the residue on a hot plate for 2 to 3 hr., overnight if convenient. Dissolve the residue in 100 ml. of water, slightly acidify with HCl, add 25 ml. of  $(\text{NH}_4)_2\text{HPO}_4$  (100 g. per l.). Cool to 15 C. (preferably use an ice bath), neutralize very slowly with  $\text{NH}_4\text{OH}$  (sp. gr. 0.90) while stirring constantly, using methyl red as an indicator, and add 10 ml. of  $\text{NH}_4\text{OH}$  in excess. Let the solution stand overnight, filter without attempting to transfer the precipitate, and wash with 1 *N*  $\text{NH}_4\text{OH}$ . Dissolve the precipitate in warm 3 *N* HCl, using the beaker from which the precipitate was filtered. Dilute to 100 ml., add 5 ml. of  $(\text{NH}_4)_2\text{HPO}_4$  (100 g. per l.), neutralize very slowly with  $\text{NH}_4\text{OH}$ , while stirring constantly, and add 5 ml. of  $\text{NH}_4\text{OH}$  in excess. Let stand for at least 4 hr., then filter through paper or asbestos which is known not to be affected by ignition with alkaline phosphates. If paper is used, it must be charred off at very low temperatures to prevent fireproofing of the paper. Ignite to  $\text{Mg}_2\text{P}_2\text{O}_7$  at 1000 to 1200 C. for 60 min. and weigh. Calculate the percentage of MgO as follows:

$$\text{MgO, per cent} = \frac{(A - B) \times 0.3621}{C} \times 100$$

where:

*A* = weight of crucible and precipitate,  
*B* = weight of crucible,  
*C* = weight of original sample, and

<sup>6</sup> The 0.1 *N*  $\text{KMnO}_4$  here used shall be standardized against National Bureau of Standards' standard sample 40c of sodium oxalate in accordance with the instructions furnished with the standard sample.

0.3621 = conversion factor from  $\text{Mg}_2\text{P}_2\text{O}_7$  to  $\text{MgO}$ .

(f) *Zinc Oxide*.—Dissolve the second sample of ash in 15 ml. of  $\text{HCl}$  (sp. gr. 1.19) in a beaker. Remove the crucible from the beaker, rinsing it thoroughly. Evaporate the solution to 5 ml. and cool. Add 10 ml. of saturated bromine water, 5 g. of  $\text{NH}_4\text{Cl}$ , 15 ml. of  $\text{NH}_4\text{OH}$  (sp. gr. 0.90), and boil vigorously for 3 min. Filter off the precipitated hydroxides, washing in four portions with 100 ml. of a solution containing 5 per cent of  $\text{NH}_4\text{Cl}$  and 2.5 per cent of  $\text{NH}_4\text{OH}$  (sp. gr. 0.90). Dilute the solution to 250 ml., heat to boiling, and add 4 drops of  $(\text{NH}_4)_2\text{S}$  to destroy oxidizing agents. Divide the solution into two exactly equal portions, dilute each to 250 ml., neutralize with  $\text{HCl}$  (sp. gr. 1.19), and add 10 ml. in excess. Titrate one portion at 75 C. with  $\text{K}_4\text{Fe}(\text{CN})_6$  using uranyl acetate as an outside indicator. The first appearance of a brown coloration on the spot plate indicates the end point. Titrate this first portion by adding 2-ml. increments of  $\text{K}_4\text{Fe}(\text{CN})_6$ . Then titrate the other portion of the solution carefully in order to get the exact end point.

Standardize the  $\text{K}_4\text{Fe}(\text{CN})_6$  against zinc of known purity by the same method as used for the determination. Each analyst should run his own standardization because of variations in color sensitivity. Blanks must be run on the standardization and on the analysis. Calculate the percentage of  $\text{ZnO}$  as follows:

$$\text{ZnO, per cent} = \frac{A \times B}{C} \times 100$$

where:

$A$  = milliliters of  $\text{K}_4\text{Fe}(\text{CN})_6$ ,

$B$  = grams of  $\text{ZnO}$  equivalent to each milliliter of  $\text{K}_4\text{Fe}(\text{CN})_6$ , and

$C$  = weight of original sample.

## Calculations

32. (a) *Acetone Extract*.—Percentage of acetone extract corrected, equals percentage of acetone extract, uncorrected, minus percentage of free sulfur, and percentage of waxy hydrocarbons. The percentage of waxy hydrocarbons equals percentage of hydrocarbons  $A$  plus percentage of hydrocarbons  $B$ . When the percentage of *organic* acetone extract is required, this shall be taken as the percentage of acetone extract, uncorrected, minus the percentage of free sulfur.

(b) *Glue*.—Multiply the percentage of rubber as compounded by 0.004 and 6.5 and call this value  $A$ . Subtract  $A$  from the percentage of glue and call this value  $B$ . Substitute  $B$  for glue in calculating the percentage of rubber hydrocarbons and then complete the calculations.

(c) *Sulfur as Antimony Trisulfide*.—Percentage of sulfur as antimony trisulfide equals percentage of total antimony multiplied by  $\frac{\text{S}_3}{\text{Sb}_2}$  equals percentage of total antimony multiplied by 0.400.

(d) *Antimony as Trisulfide*.—Percentage of total antimony as trisulfide equals percentage of total antimony multiplied by  $\frac{\text{Sb}_2\text{S}_3}{\text{Sb}_2}$  equals percentage of total antimony multiplied by 1.400.

(e) *Total Sulfur, Corrected*.—Percentage of total sulfur corrected equals percentage of total sulfur minus percentage of sulfur as barium sulfate minus percentage of sulfur as antimony trisulfide. When barium carbonate is found, the percentage of sulfur necessary to convert it to sulfate must be added to the total sulfur, corrected.

(f) *Ash, Corrected*.—Percentage of ash corrected equals percentage of ash minus percentage of sulfur in ash plus per-

centage of sulfur as barium sulfate minus percentage of antimony in ash, calculated to  $\text{Sb}_2\text{O}_3$ .

(g) *Rubber Hydrocarbon*.—Percentage of rubber hydrocarbon equals  $94/97(100 - C)$  where  $C$  equals the sum of the following constituents, except when it is determined by the Joint Rubber Insulation Committee's Procedure (Section 28):

Organic acetone extract  
Chloroform extract  
Alcoholic-potash extract  
Corrected total sulfur  
Ash, corrected  
Carbon  
Glue  
Total antimony as trisulfide

#### Rubber as Compounded

33. The percentage of rubber hydrocarbon plus 6 per cent of its weight is taken as "rubber as compounded," except when the sum of the percentages of the acetone extract corrected, chloroform extract, and alcoholic-potash extract is less than the figure represented by the arbitrary 6 per cent, as in many high-grade compounds. Percentage of rubber as compounded is then equal to the percentage of rubber hydrocarbon plus the sum of the percentages of the acetone extract corrected, the chloroform extract, and the alcoholic-potash extract.

$$\text{Rubber by volume, per cent} = \frac{R \times G}{0.911}$$

$$\text{Ratio of organic acetone extract} = \frac{A}{R} \times 100$$

$$\text{Ratio of sulfur to rubber} = \frac{S}{R} \times 100$$

where:

$R$  = the percentage of rubber as compounded,  
 $G$  = the specific gravity of the compound,

0.911 = the assumed specific gravity of rubber,  
 $A$  = the percentage of organic acetone extract, and  
 $S$  = the percentage total sulfur, corrected.

#### Report

34. The report shall include the following:

- (a) Percentage of acetone extract, corrected,
- (b) Percentage of waxy hydrocarbons,
- (c) Percentage of chloroform extract,
- (d) Percentage of alcoholic-potash extract,
- (e) Percentage of free sulfur,
- (f) Percentage of total sulfur, corrected,
- (g) Percentage of ash, corrected,
- (h) Percentage of cellulose,
- (i) Percentage of sulfur as barium sulfate,
- (j) Percentage of total antimony,
- (k) Percentage of sulfur as antimony trisulfide,
- (l) Percentage of carbon,
- (m) Percentage of glue,
- (n) Percentage of rubber hydrocarbon,
- (o) Percentage of rubber, as compounded,
- (p) Percentage of rubber by volume,
- (q) Ratio of acetone extract to rubber as compounded,
- (r) Ratio of sulfur to rubber as compounded, and
- (s) Specific gravity.

#### B. RUBBER SOLVENT METHOD

##### Nature of Test

35. (a) Solvents for vulcanized rubber have been suggested from time to time in attempts to isolate the compounding ingredients in rubber goods by methods

which would avoid the thermal decomposition incident to the ash method. Aniline, terebene, kerosine, toluene, and cymene have been used with success. For the most part, filtration is slow with these solvents.

(b) In the methods given in Sections 36 to 38, the rubber solvent consists of a mixture of 300-deg. mineral seal oils from two or more sources.<sup>6a</sup> Before use the mixed oils are passed through a column of fuller's earth contained in a glass tube 3 ft. in length and 1½ in. in diameter. The filtered mixture is practically colorless. The rubber hydrocarbon in vulcanized soft rubber compounds is dissolved by the solvent in about 2 hr. at 130 to 150 C. The subsequent operations incident to isolating the compounding ingredients are conducted as readily as with an aqueous solution. The solvent is recommended because of completeness of separation and the rapidity with which it may be filtered. It has been determined empirically that the colloidal solution of rubber formed does not have the disadvantage of slow filtration which is typical of some other solvents of vulcanized rubber.

(c) After the mixture of mineral oils has acted on a rubber compound, the rubber is dissolved and the fillers remain for some time in suspension in the solution. On continued heating the fillers settle to the bottom of the container. In mixtures of asbestos fibers and rubber compound, such as are present in compressed asbestos sheet packing, it is possible by the above difference in behavior to separate the rubber and rubber compound ingredients from the asbestos fibers. While the fillers are still in suspension the solution is poured through a sieve, which retains the fibers. A 177-micron (No. 80) sieve<sup>3</sup> is recommended for the purpose. Subsequently, by con-

tinued heating the rubber and compounding ingredients may be separated. Any asbestos present as asbestine is separated with the compounding ingredients. In the case of rubber goods prepared from new rubber, a small amount of undissolved material is obtained for which a correction must be made.

NOTE.—See the statement regarding decomposable fillers, Section 8 (d).

#### Procedure

36. Weigh two portions "a" and "b" of from 0.5 to 1 g. each of the finely-ground sample. Extract with a mixture of 32 per cent of acetone and 68 per cent of chloroform by volume for a minimum of 8 hr.<sup>7</sup> If the liquid in the extraction apparatus is still colored at the end of this time, continue the extraction. Remove the samples and put each into a 150-ml. lipped assay flask, add 20 to 25 ml. of the oil (see Section 3 (i)), cover with a watch glass and heat in an air bath at a temperature of 150 to 155 C. until solution appears complete, and then continue the heating for 15 to 30 min. more. Solution may be considered complete when the rubber colloid has been broken down and the oil seems quite clear. Remove the flasks from the air bath, cool to about 110 C., and add in a small stream 10 to 15 ml. of benzene, while mixing thoroughly; allow to cool and then dilute with sufficient petroleum ether to fill each flask to within about 2 cm. of the top. Mix thoroughly, cover the flasks to prevent evaporation and allow the mixture to stand overnight.

37. *Treatment of Portion "a."*—(a) Prepare a Gooch crucible with finely divided asbestos that has been previously treated with strong NaOH solution and HCl (sp. gr. 1.19) and washed well with water. Ignite the crucible, cool, and

<sup>6a</sup> Editorially revised in February, 1945.

<sup>7</sup> National Bureau of Standards *Technologic Paper No. 162*; also *Rubber Age and Tire News*, pp. 445-447 (1920).

weigh; call this weight *c*. Filter the mixture by decantation through the crucible, using suction; wash well with petroleum ether, followed by warm acetone, and by a warm mixture of equal volumes of acetone and chloroform if the filtrate is dark. Remove as much as possible of the organic residue by washing; finally, wash with hot alcohol. A portion of the fillers will remain in the flask. Dry the crucible and flask with their contents for 1 hr. at a temperature of 105 to 110 C. Cool, and weigh. Call the weight of the flask and contents *d*, and the weight of the crucible and contents *e*.

(b) Remove the acid-soluble compounding ingredients from the flask and Gooch crucible, collecting the solution in a 400-ml. beaker, by adding to the flask and crucible a few milliliters of boiling alcohol. Allow to soak for 2 or 3 min. and then wash two or three times with boiling water; let the flask cool, add 10 ml. of HCl (sp. gr. 1.19) and swirl the flask to bring the acid in contact with the compounding ingredients. Pour the acid from the flask into the crucible and let it stand until no more bubbles rise through the liquid. If carbonates are present, there is danger of loss by excessive frothing. This can be prevented by first adding a few drops of the acid to the crucible and sucking it through the pad. After the violent action has ceased, add the remainder of the 10 ml. of acid. When no more gas is evolved, draw the acid through the pad and again wash with 20 ml. of acid, adding a little at a time. Wash well with hot water, and transfer as much as possible of the residue remaining in the flask to the asbestos pad. If by qualitative tests the sample was found to contain antimony, save the filtrate and washings for treatment as described in Paragraph (c). Dry the flask and crucible for 1 hr. at 105 to 110 C., cool, and weigh. Call the weight of

the flask *f*, and that of the crucible containing the organic residue and acid-insoluble fillers *h*. Burn the organic residue from the asbestos pad by igniting in a furnace at a temperature of 700 C., cool, and weigh. Call this weight *k*. If the sample contains barytes, save the contents of the crucible for treatment as described in Paragraph (d).

(c) *Determination of Sulfur Present as Antimony Trisulfide.*<sup>8</sup>—Dilute the filtrate in the beaker (see Paragraph (b)) to about 250 ml. and precipitate the antimony with H<sub>2</sub>S. Filter off the antimony sulfide and wash the precipitate with H<sub>2</sub>S water to which a little HCl has been added. Transfer the filter paper containing the antimony precipitate to a Kjeldahl flask, and add 12 ml. of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84), and 5 g. of potassium sulfate. Heat the solution gently until it is colorless, dilute to 100 ml. and add 1 to 2 g. of sodium sulfite. Boil until the sulfur dioxide is driven out. Determine antimony from this point as described in Section 21. Calculate the antimony present to antimony trisulfide and express the sulfur present as antimony trisulfide in percentage of the sample and call this *L*.

(d) *Determination of Barytes.*—Transfer the contents of the crucible (see Paragraph (b)) to a 50-ml. porcelain crucible and fuse with 5 g. of a mixture of equal parts of sodium carbonate and nitrate. Stir well during the fusion. Cool the crucible, put it into a 250-ml. beaker, cover with distilled water and proceed from this point exactly as in the analysis of barium sulfate, Section 20.

$$B = m = \frac{100 \times \text{wt. of BaSO}_4 \times 0.1373}{\text{wt. of sample "a"}}$$

where *B* = the percentage of sulfur present as barytes.

<sup>8</sup> S. Collier, M. Levin and J. A. Scherrer, "Determination of Antimony in Rubber Goods," *India Rubber World*, Vol. 64, p. 580 (1921).

38. *Treatment of Portion "b" for Sulfur in Compounding Ingredients.*—Treat portion "b" as described in Section 37 (a), but omit the weighing of the crucible. After the pad has been dried, transfer it to the flask. The last traces of the fillers can be removed from the sides of the crucible with wads of absorbent cotton moistened with a little warm water. Add to the fillers about 10 ml. of bromine water, and swirl the flask to moisten the contents. Add 20 ml. of  $\text{HNO}_3$  (sp. gr. 1.42) saturated with bromine. Allow to stand for 15 min. and then heat on the steam bath for 1 hr. Transfer the contents of the flask to a 75-ml. crucible and evaporate to dryness. Complete the determination of sulfur by the method described in Section 15. Let the value for the percentage of sulfur in compounding ingredients be represented by  $n$ .

#### Calculations

39. (a) Calculate the percentage of total inorganic compounding ingredients, corrected, from Eq. (1).

$$\frac{\text{Total inorganic compounding ingredients corrected}}{\text{wt. of sample}} = \frac{100[(e + d + k) - (c + f + h)]}{\text{wt. of sample}} + (m + L) - n \dots (1)$$

(b) To determine the percentage of rubber hydrocarbon subtract from 100 the sum of the percentages of acetone, chloroform, and alcoholic-potash extracts, free carbon, glue, total sulfur corrected, and total inorganic compounding ingredients corrected.

#### C. SHORT PROCEDURE

##### Scope

40. The short procedure shall be used only for the analysis of simple rubber compounds consisting of rubber, sulfur and undecomposable compounding in-

gredients as shown by the preliminary examination of the sample described in Section 7 (a) to (f), inclusive, or as specified.

##### Acetone Extract

41. The acetone extract shall be determined as described in Section 11.

##### Free Sulfur

42. The free sulfur shall be determined as described in Section 16.

##### Alcoholic Potash Extract

43. The alcoholic potash extract shall be determined as described in Section 13.

##### Total Sulfur

44. The total sulfur shall be determined as described in Section 15.

##### Ash

45. The ash shall be determined as described in Section 17.

##### Calculations

46. The percentage of rubber shall be

considered to be the difference between 100 and the sum of the total sulfur, ash, free carbon, and similar ingredients. If the alcoholic-potash extract is over 2 per cent of the rubber as first calculated, subtract this excess also from the rubber. The organic-acetone extract shall be obtained by taking the difference between the total acetone extract and the free sulfur. The organic-acetone extract, free sulfur, total sulfur, and alcoholic-potash extract shall be figured on the amount of gum as found by the above procedure.

##### Check Analyses

47. Duplicate determinations when

required shall check within the following limits, expressed as percentages of the gum present, as found by analysis, except as stated:

	Check Within, per cent
Organic-acetone extract.....	0.20
Free sulfur.....	0.10
Total sulfur.....	0.10
Alcoholic-potash extract.....	0.10
Ash calculated on total compound...	0.25

#### D. DIRECT DETERMINATION OF RUBBER HYDROCARBON<sup>9</sup>

##### Nature of Test

48. This procedure covers the determination of rubber hydrocarbon in crude and vulcanized rubber compounds, balata, and in mixtures of rubber with certain rubber-like materials (Note). The procedure consists essentially of quantitatively oxidizing the rubber hydrocarbon by means of digestion in chromic acid solution, after which the acetic acid formed is separated by distillation, and determined by titration of the distillate after carbon dioxide has been removed by aeration. This procedure is also applicable to reclaimed rubber, but it has been found to give consistently lower than previously accepted estimates of the rubber hydrocarbon content of reclaimed rubber.

NOTE.—Limitations as to types of materials analyzed by this procedure and interference from the usual compounding ingredients are summarized in Tables I and II.

##### Apparatus

49. The apparatus shall consist of the following:

(a) *Digestion and Distillation Assembly*.—The digestion and distillation apparatus shown in Fig. 5 may be conveniently assembled on a ring stand with a tripod foot. The use of rubber connections shall be avoided where they might come into contact with the digestion mixture.

<sup>9</sup> V. L. Burger, W. E. Donaldson, and J. A. Baty, "A Direct Determination of Rubber Hydrocarbon, Chromic Acid Oxidation Method," ASTM BULLETIN, No. 120, January, 1943, p. 23.

(b) *Aeration Assembly*.—An aeration assembly (see Fig. 5) containing a capillary tube, *J*, which when connected to the vacuum line will maintain through the receiving flask an air flow of approximately 2 l. per min. If the vacuum is less than 30 mm. of mercury a capillary

TABLE I.—DEGREE OF INTERFERENCE OF RUBBER COMPOUNDING INGREDIENTS.

Compounding Ingredient	Interference
Combined sulfur.....	none in normal soft cures
Carbon black.....	none as tested in tread stocks
Cellulose.....	negligible, 2 per cent or less of its weight reacts as if it were rubber hydrocarbon
Asphaltic hydrocarbon (mineral rubber).....	removed by acetone and chloroform extraction. If not extracted, approximately 45 per cent of its weight reacts as if it were rubber hydrocarbon
Factice, brown.....	negligible, after acetone and chloroform extraction

TABLE II.—BEHAVIOR OF RUBBER-LIKE MATERIALS IN CHROMIC ACID OXIDATION PROCEDURE.

Material	Value Obtained
Hard rubber.....	approximately 50 per cent of its weight reacts as if it were rubber hydrocarbon
Balata.....	approximately equivalent to rubber
Thiokol RD.....	approximately 18 per cent of its weight reacts as if it were rubber hydrocarbon
Perbunan.....	approximately 1.5 to 2 per cent of its weight reacts as if it were rubber hydrocarbon
Buna S.....	approximately 3 per cent of its weight reacts as if it were rubber hydrocarbon
Vistenex.....	virtually unattacked
Neoprene GN.....	approximately 3 per cent of its weight reacts as if it were rubber hydrocarbon if a modification of the procedure is used to avoid the interference of chlorine <sup>a</sup>

<sup>a</sup> This modification consists of adding neutral potassium iodide to the distillate after aeration and titrating any iodine which may be released with neutral sodium thiosulfate before proceeding with the titration with 0.1000 *N* NaOH.

tube approximately 10 cm. in length with an 0.75-mm. bore will maintain the required air flow. Since it is essential that the aeration be maintained at a rate within 10 to 20 per cent of 2 l. per min., each capillary shall be tested before use. The following method may be used: Invert a graduate over a beaker filled



with water and evacuate the air through the capillary by means of a tube extending up into the graduate. The rate of air flow will be the same as the rate at which the water fills the graduate.

### Reagents

50. (a) *Chromic Acid Digestion Mixture*.—Dissolve 200 g. of reagent grade  $\text{CrO}_3$  in 500 ml. of distilled water, add 150 ml. of reagent grade  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84), and mix well.

(b) *Standard Sodium Hydroxide Solution* (0.1000 N).

(c) *Alcoholic Phenolphthalein Indicator Solution* (10 g. per l.).—Dissolve 10 g. of U.S.P. phenolphthalein in 500 ml. of U.S.P. ethanol, and dilute to 1 liter.

### Procedure

51. (a) *Vulcanized Rubber*.—Weigh a sufficient amount of the sample sheeted to a thickness of 0.5 mm. (Note 1), to contain approximately 0.3 g. of rubber hydrocarbon. Wrap the sample loosely in filter paper, and extract with acetone for at least 8 hr. as described in Section 11. After the acetone extraction, extract the sample with chloroform for 4 hr. as described in Section 12. Dry in an oven at 100 C. for 1 hr.

(b) *Unvulcanized or Reclaimed Rubber*.—Weigh a sufficient amount of the sample sheeted to a thickness of 0.5 mm. (Note 1), to contain approximately 0.3 g. of rubber hydrocarbon. If the material is unvulcanized or reclaimed rubber, suspected of containing mineral rubber or factice, vulcanize the material by a simple known recipe, and after a sample of the vulcanizate has been prepared, extract it with acetone and chloroform (Sections 11 and 12) (Note 2). Dry in an oven at 100 C. for 1 hr.

(c) Place 700 to 900 ml. of distilled water in the steam-generating flask, A.

Place a few milliliters of water, sufficient to cover the end of the adapter, in the receiving flask, H. Mark the outside of the digestion flask, C, at a point indicating the liquid level when the flask contains 75 ml. Transfer  $50 \pm 1$  ml. of the chromic acid digestion mixture in the digestion flask. Lift the steam tube, B, and insert the sample extracted in accordance with Paragraph (a) or (b) into the digestion flask (Note 3). Replace the steam tube and tighten the connection. Heat the beaker of water surrounding the digestion flask to boiling, continue boiling for 1 hr., and remove the burner and beaker.

(d) During the digestion period, heat the steam-generating flask, A, with the stopper removed, until the contents are boiling. At the end of the digestion period, replace the stopper and outlet tube in the mouth of the steam-generating flask, adjust the burner to maximum heat, and pass the steam through the digestion flask, C. When the volume of the liquid in the digestion flask is increased to approximately 75 ml., place a small flame under the flask, keeping it at a point which will maintain the volume in the digestion flask at 75 ml. Continue the distillation until 500 ml. have been collected in the receiving flask, H. Remove the burners, immediately remove the receiving flask and the adapter, G, and rinse the adapter with water from a wash bottle, catching the washings in the receiving flask.

(e) Adjust the temperature of the liquid in the receiving flask to  $25 \pm 5$  C., insert the aeration assembly into the receiving flask, and attach to a vacuum line. Draw a stream of air through the liquid for 30 min. at a rate of approximately 2 l. per min. (Note 4). Remove the rubber tubing and loosen the two-hole stopper. Rinse the stopper and glass tubing with water from a wash bottle, catching the rinsings in the

receiving flask. Add alcoholic phenolphthalein indicator solution (10 g. per l.) to the receiving flask, and titrate with 0.1000 *N* NaOH.

(f) *Blank*.—Make a blank determination, following the procedure described in Paragraphs (c), (d), and (e) and using the same amounts of all re-

mended. Variation of any of these factors may lead to erroneous analytical results.

### Calculation

52. Calculate the percentage of rubber hydrocarbon as follows (Note):

$$\text{Rubber hydrocarbon, per cent} = \frac{0.908(A-B)}{C}$$

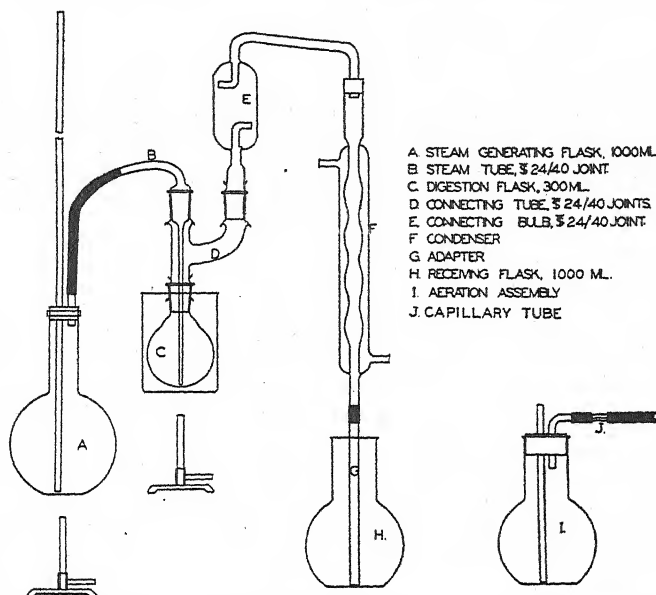


FIG. 5.—Apparatus for Direct Determination of Rubber Hydrocarbon.

gents. The blank should not exceed 0.2 to 0.3 ml.

NOTE 1.—The sample must be sheeted to a thickness of 0.5 mm. or less, since some materials, such as crude rubber, may not otherwise be completely attacked by the oxidation mixture in the time prescribed for the digestion.

NOTE 2.—Poorly vulcanized rubbers must not be extracted with chloroform.

NOTE 3.—It is not necessary to remove the filter paper quantitatively from the sample before transferring it to the digestion flask, since the interference of small amounts of cellulose is negligible.

NOTE 4: *Caution*.—The rates of loss of carbon dioxide and of acetic acid during aeration have been investigated for the temperature range, type of apparatus, and rate of air flow recom-

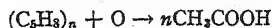
where:

*A* = milliliters of NaOH solution required to titrate the sample,

*B* = milliliters of NaOH solution required to titrate the blank, and

*C* = weight of sheeted sample in grams.

NOTE.—This calculation is based on the observation that a 75 per cent yield is always obtained in the reaction:



using purified rubber. To calculate the results on the basis of crude rubber, a suitable correction for nonrubber constituents of crude rubber is necessary.

**Accuracy**

53. Results may be expected to be accurate within 2 per cent based on the weight of the sample. Results are usually consistent to 0.5 per cent or less.

**E. DETERMINATION OF COPPER AND MANGANESE IN CRUDE RUBBER****Reagents**

54. (a) *C.P. Ammonium Hydroxide* (sp. gr. 0.90).

(b) *C.P. Fuming Nitric Acid*.

(c) *C.P. Phosphoric Acid* (85 per cent).

(d) *C.P. Sulfuric Acid* (sp. gr. 1.84).

(e) *Standard Copper Sulfate Solution* (1 ml. = 0.00001 g. Cu).—Dissolve 0.3928 g. of c.p.  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , which contains 0.1000 g. of copper, in 250 ml. of copper-free water in a volumetric flask. Pipette 25 ml. of this solution into a 1-liter volumetric flask and dilute to volume with copper-free water. This final solution is the standard and will contain 0.00001 g. of copper per milliliter.

(f) *Hydrogen Peroxide*.—A solution of hydrogen peroxide, approximately 30 per cent by volume, essentially free of copper and manganese.

(g) *Standard Potassium Permanganate Solution* (1 ml. = 0.0001 g. Mn).—Prepare a diluted solution of  $\text{KMnO}_4$  by diluting to 500 ml. in a volumetric flask, a quantity of standard  $\text{KMnO}_4$  solution in accordance with the following formula:

$$A = \frac{4.551}{N}$$

where:

$A$  = number of milliliters of standard  $\text{KMnO}_4$  required, and

$N$  = normality of the standard  $\text{KMnO}_4$  used.

This solution contains 0.0001 g. of manganese per milliliter. Do not keep this solution for more than one week.

(h) *C.P. Potassium Periodate*.

(i) *Sodium Diethyldithiocarbamate Solution*.—Dissolve 1.0 g. of the salt in a liter of copper-free water. Place in an amber bottle and protect from strong light.

(j) *Gum Arabic Solution* (5 per cent).—Prepare a 5 per cent solution of gum arabic in distilled water.

**Blank Determinations**

55. Run simultaneously, with each set of determinations, blank determinations using the same quantities of the same reagents. All reagents, including the distilled water shall be as free as possible from copper and manganese.

**Destruction of Organic Matter**

56. Accurately weigh approximately 5 g. of the sample and transfer to a 500-ml. Kjeldahl flask. Add carefully 20 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) and one or two glass beads, place the flask on a digestion rack, and heat slowly until the mixture boils. Continue to boil gently until complete charring and disintegration of the organic matter have occurred (boiling for about 15 to 20 min. is generally required). As  $\text{H}_2\text{SO}_4$  is consumed in the oxidation, add more in 5-ml. portions, when needed, to maintain the volume at about 20 ml. When charring is complete, allow to cool and add carefully, in small portions, 5 ml. of fuming nitric acid. If a strong reaction occurs, stop the addition and swirl the contents of the flask until the reaction subsides, then carefully continue the addition. Heat the mixture with a low flame until the brown fumes have disappeared, boil vigorously for a few minutes, and then cool. Repeat this process until two successive treatments with the nitric acid produce no decrease in color (three 5-ml. portions of nitric acid are generally sufficient). While agitating,

dilute the contents of the flask with 100 ml. of distilled water. Boil the solution down to strong fumes of sulfur trioxide and cool. This hydrolyzes the nitrosyl sulfuric acid and drives off oxides of nitrogen. If a yellow color is present in the solution at this point it is usually indicative of the presence of either iron or undigested organic matter, and the solution should be treated as follows: Carefully add 5 ml. of hydrogen peroxide. Heat the mixture with a low flame to strong fumes of sulfur trioxide, boil vigorously for a few minutes, and cool. Repeat this process until two successive treatments with hydrogen peroxide produce no decrease in color (two or three 5-ml. portions are generally sufficient). While agitating, dilute the contents of the flask with 100 ml. of distilled water. Boil the solution down to strong fumes of sulfur trioxide and then cool. This will remove any excess of hydrogen peroxide. Dilute the solution with 100 ml. of distilled water. If the solution is clear, transfer to a 250-ml. volumetric flask, dilute to volume, and mix well. If the solution contains insoluble matter, heat to boiling and filter hot, by gravity, through No. 1 Whatman filter paper or equivalent. Wash the filter well with hot distilled water, dilute to volume in a 250-ml. volumetric flask, and mix well. Aliquots of this solution are used for the determination of copper, and of manganese if it is present in sufficient quantity.

### Copper

57. (a) Pipette a suitable aliquot, usually 25 ml. of the solution, into a small Erlenmeyer flask or beaker. Drop in a small piece of litmus paper as indicator and make just alkaline with  $\text{NH}_4\text{OH}$ . Add 1 to 2 ml. excess  $\text{NH}_4\text{OH}$  (sp. gr. 0.90), heat to boiling, and place on a steam bath until coagulation and precipitation of iron hydroxide are

complete. If aluminum is known to be present, allow to stand on the steam bath for at least 1 hr. to insure complete precipitation of aluminum hydroxide. Filter by gravity, through No. 1 Whatman filter paper or equivalent, into a 100-ml. Nessler tube, and wash the filter with two or three small portions of hot distilled water. To the solution in the Nessler tube, add 1 ml. of gum arabic solution (5 per cent), 10 ml. of c.p.  $\text{NH}_4\text{OH}$  (sp. gr. 0.90), and 10 ml. of sodium diethyldithiocarbamate solution in the order named. Dilute to the mark and mix well. To a second tube containing a similar aliquot of a blank solution carried through the entire analysis in the same manner as the sample, add equal amounts of the same reagents, dilute to about 90-ml., and mix. Titrate into the second tube with the standard copper solution from a 10-ml. burette (preferably graduated in twentieths of a milliliter) until its color matches that of the sample when diluted to the same volume. Mix well after each addition of copper solution. If the color in the sample tube is too deep for comparison, a smaller aliquot portion of the solution from the acid digestion must be used. If the color in the sample tube is too light for a good comparison, the amount of copper present (on the basis of a 5-g. sample) is below the accuracy of the method. Best results will be obtained when the aliquot contains copper equivalent to 1 to 5 ml. of the standard copper solution.

(b) In case the solution in the tube containing the sample is turbid, some interfering substance is present, and a modified or different procedure will be necessary.

(c) *Calculation.*—Calculate the percentage of copper as follows:

$$\text{Copper, per cent} = \frac{\text{ml. of standard copper solution} \times 0.001}{\text{wt. of sample in aliquot}}$$

**Manganese**

58. (a) Transfer a suitable aliquot, usually the balance of the solution in the 250-ml. volumetric flask (Section 56), to a 250-ml. beaker and evaporate to about 75-ml. volume. (If the manganese content is very low a separate sample should be used and the whole solution from the acid digestion should be evaporated for the comparison.) Add 10 ml. of c.p. phosphoric acid (85 per cent) to decolorize iron if present. Sprinkle in 0.5 g. of potassium periodate and bring the solution to a boil. Cool slightly, sprinkle in another small portion of potassium periodate (about 0.1 g.) and boil again. When the color seems to have developed to a maximum, place the beaker on a steam bath and keep hot for 15 min. Should there be any doubt about the completeness of the reaction, add more potassium periodate. After the sample has stood on the steam bath for 15 min. remove it, and allow to cool.

If the color is too deep for a good comparison, wash into a 250-ml. volumetric flask and dilute to volume with distilled water. Place a suitable aliquot (or the whole sample) in a 100-ml. Nessler tube and dilute to the mark with distilled water. Place a similar aliquot of a blank solution, carried through the entire analysis in the same manner as the sample, in a second tube and dilute almost to the mark. Titrate into this second tube with the standard manganese solution from a 10-ml. burette (preferably graduated in twentieths of a milliliter) until its color matches that of the sample when diluted to the same volume. Mix the contents of the tube by pouring into a small beaker and stirring, not by shaking in the stoppered tube or using the hand as a stopper.

(b) *Calculation.*—Calculate the percentage of manganese as follows:

$$\text{Manganese, per cent} = \frac{\text{ml. of standard manganese solution} \times 0.01}{\text{wt. of sample in aliquot}}$$

# *Tentative Methods of*

## IDENTIFICATION AND QUANTITATIVE ANALYSIS OF SYNTHETIC ELASTOMERS<sup>1</sup>



A.S.T.M. Designation: D 833 - 46 T

ISSUED, 1945; REVISED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods of identification and quantitative analysis of synthetic elastomers<sup>3</sup> are classified according to the following three divisions:

- A. Identification of Elastomers,
- B. Quantitative Analysis of Elastomers, and
- C. Determination of Compounding Ingredients.

Method A includes a semi-routine qualitative scheme with confirmatory tests. Method B includes methods of quantitatively analyzing for individual elastomers present in an unknown compound. Method C covers the determination of compounding ingredients in synthetic rubber compounds.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Administrative Committee on Standards, September 9, 1946.

<sup>3</sup> For information concerning the development of these methods, the following references may be consulted:

H. P. Burchfield, "Identification of Natural and Synthetic Rubbers," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 16, p. 424 (1944).

H. P. Burchfield, "Qualitative Spot Tests for Rubber Polymers," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 17, p. 806 (1945).

"Identification and Estimation of Natural and Synthetic Rubbers," British Ministry of Supply, Admiralty, Ministry of Aircraft Production (1944).

### Definitions

2. (a) *Synthetic Elastomer*.—Any of the synthetic rubber-like materials and some synthetic resins which occasionally occur mixed with these rubber-like materials.

NOTE.—This definition is intentionally made broader than is usual in the strictly scientific sense.

(b) *Chloroprene*.—Polymers of chloroprene with or without co-monomers present.

(c) *Styrene Rubbers*.—Styrene-butadiene copolymers with or without other co-monomers present.

(d) *Nitrile Rubbers*.—Butadiene-acrylonitrile copolymers with or without other co-monomers present.

(e) *Polyisobutylene Rubbers*.—Polymerized isobutylene with or without co-monomers present.

(f) *Thioplasts*.—Any of the polysulfide rubbers consisting of organic radicals linked through sulfur.

(g) *Polyvinyl Plastics*.—Polyvinyl chloride, polyvinyl acetate, or mix-

tures of these with true elastomers.

(h) The descriptions of terms given in Section 2 of the Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297)<sup>4</sup> shall also apply to the terms appearing in these methods.

### Reagents

3. Reagents shall conform to the requirements prescribed in A.S.T.M. Methods D 297.

### A. IDENTIFICATION OF ELASTOMERS

#### Application

4. This method is suitable for the identification of natural rubber and the common synthetic elastomers and polyvinyl resins when each is present alone in a compound. In some cases, components of mixtures may be identified. Supplementary tests based on the experience of the operator may be employed to advantage in many cases.

#### Apparatus

5. The apparatus shall consist of the following:

(a) *Distillation Apparatus*.—Test tubes, 10 by 75 mm., equipped with a glass condensing tube about 4 mm. in outside diameter attached to the test tube by means of a cork stopper. The condensing tube shall be bent at least 90 deg. and shall extend about 100 mm. beyond the bend.

(b) *Receiver*.—Test tubes, 10 by 75 mm., for collecting distillate.

#### Reagents

6. (a) *Solution I*.—Dissolve 1.0 g. of *p*-dimethylaminobenzaldehyde and 0.01 g. of hydroquinone in 100 ml. of absolute methanol. Add 5 ml. of HCl (sp. gr. 1.18) and 10 ml. of ethylene glycol. Adjust the specific gravity to 0.851 at

25/4 C. by the addition of a calculated amount of methanol or ethylene glycol. The reagent is stable over a period of several months when stored in a brown bottle.

(b) *Solution II*.—Dissolve 2.00 g. of sodium citrate ( $2\text{Na}_3\text{C}_6\text{H}_5\text{O}_7 \cdot 11\text{H}_2\text{O}$ ), 0.2 g. of citric acid, and 0.03 g. of Metanil yellow in 500 ml. of distilled water.

(c) *Chloroprene-Nitrile Spot Test Papers and Wetting Solution*.—Dissolve 2.0 g. of cupric acetate and 0.25 g. of Metanil yellow in 500 ml. of methanol. Impregnate filter paper squares with the solution, dry, and cut into strips. To prepare the wetting solution, dissolve 2.5 g. of benzidine dihydrochloride in a mixture of 500 ml. of methanol and 500 ml. of water. Add 10 ml. of 0.1 per cent aqueous solution of hydroquinone. Store in a brown bottle. A precipitate, which usually forms on standing, does not affect the efficiency of the solution. If the solution is protected from light and air it can be used for several months.

(d) *Polyisobutylene Spot Test Papers and Wetting Solution*.—Use blank filter paper strips. To prepare the wetting solution, add 5.0 g. of yellow mercuric oxide to a mixture of 15 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) and 80 ml. of water. Bring to a boil, and continue heating until the oxide dissolves. Cool and dilute to 100 ml. with water.

(e) *Rubber-Styrene Spot Test Papers and Wetting Solution*.—Impregnate filter paper squares with a solution of 3 g. of *p*-dimethylaminobenzaldehyde and 0.05 g. of hydroquinone in 100 ml. of ethyl ether. Dry and cut into strips. Papers stored in brown glass bottles are stable for several weeks but will lose their efficiency if stored in light. To prepare the wetting solution, dissolve 30 g. of trichloroacetic acid in isopropanol and dilute to 100 ml. with isopropanol. Contact of this reagent with the skin should be avoided.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



### Identification by Pyrolysis Products

7. Strip the rubber from any adhering fabric. Place 0.5 g. of the sample in a test tube and attach the side arm. Heat with a microburner or very small bunsen flame until the sample begins to decompose. When vapors appear at the mouth of the side arm, immerse the end beneath the surface of 1.5 ml. of Solution II contained in the receiver test tube. After it is evident whether a color change will take place or not, remove the tube and continue the distillation into 1.5 ml. of Solution I in another test tube. Permit

TABLE I.—PYROLYSIS TEST.

Material	Solution I		Solution II
	Initial Color	Color After Heating	
Blank .....	pale yellow	pale yellow	green
Polyvinyl chloride.....	yellow	yellow	red
Chloroprene ..	yellow	pale yellow green	red
Nitrile .....	orange red	red	green
Chloroprene-nitrile.....	orange red	red	yellow to red
Styrene .....	yellow green	green	green
Natural rubber.....	brown	violet blue	green
50 styrene-50 rubber.....	olive green	green blue	green
Polyisobutylene.....	yellow (drop- let floats)	pale blue green	green
Polyvinyl acetate.....	yellow	pale yellow green	yellow

the receivers to cool, and shake. Note whether the drops sink or float in Solution I and note the color changes in both solutions. Transfer Solution I to a 16 by 150-mm. test tube and add 5 ml. of absolute methanol. Heat on a water bath at 100 C. for 3 min. and note the color that develops. Record all observations, and classify the material by means of Table I.

### Identification by Spot Tests

8. (a) Wet a strip of the test paper with the corresponding impregnating solution (Section 6 (c), (d), and (e)), and hold it in a parallel position about 5 mm.

above the surface of a heating element which is pressed against the rubber. The heating element may be an electrically heated knife or iron or an iron or file tip heated by a flame. It should be hot enough to cause dense fumes of pyrolysis product to be produced but not sufficiently hot to ignite the rubber. With vigorous evolution of fumes a test may be carried out in 4 to 6 sec. Care shall be taken to obtain a good color response on the side facing the fumes without scorching the paper or the impregnating materials.

(b) The colors obtained with the elastomers are summarized in Table II. The chloroprene-nitrile elastomer test shall be carried out first. If both of

TABLE II.—SPOT TEST.

Elastomer	Chloroprene-Nitrile Test	Polyisobutylene Test	Rubber-Styrene Test
Chloroprene .....	red	blank <sup>a</sup>	green
Nitrile .....	green	pale brown	yellow green
Chloroprene-nitrile mixture.....	red/green	pale brown	green
Polyisobutylene ..	blank <sup>a</sup>	yellow	pale lavender
Natural rubber ..	blank <sup>a</sup>	brown	blue
Styrene .....	blank <sup>a</sup>	brown	blue green

<sup>a</sup> Blank color tests may be pale brown rather than colorless.

these elastomers are absent, the other tests will normally be successful.

(c) *Mixtures of Elastomers.*—Chloroprene-nitrile elastomer mixtures containing more than about 30 per cent chloroprene type give a green color on the wet portion of the test paper and a red color on the dry portion. Less than 30 per cent chloroprene type usually cannot be detected in this mixture. Chloroprene type will not normally mask a natural or polyisobutylene type elastomer test but may mask styrene type. Nitrile elastomer will mask a styrene elastomer and may cause some difficulty in detecting natural rubber. Polyisobutylene elastomer normally can be detected in the presence of moderate amounts of the other four elastomers.

The chloroprene-nitrile test is not masked by the presence of the other three elastomers. Styrene elastomer is quite difficult to detect unless alone or with only small quantities of other elastomers. Styrene elastomers and natural rubber together can both be identified only in the range of 50-50 mixtures. It must be realized by the operator that the spot tests alone are not infallible for detecting all elastomers present in a mixture. However, a combination of these tests with the confirmatory tests should enable an operator to identify essentially all mixtures of elastomers.

### Confirmatory Tests

9. (a) Confirm the identification of elastomers made in accordance with Section 7 or 8 by means of the tests described in Paragraphs (b) to (f).

(b) *Chloroprene Polymers*.—Chloroprene polymers may be distinguished from the saturated polyvinyl type by the following iodine test: Shake a 0.2-g. sample of the elastomer with 2 ml. of iodine solution (0.2 g. of iodine per liter of  $\text{CCl}_4$ ). If the violet color fades noticeably in 2 to 3 min., a chloroprene polymer is indicated. Further confirmation of the presence of chlorine-containing polymers, particularly in the presence of much nitrile polymer, may be obtained by burning the elastomer in contact with a clean copper wire. A persistent green flame indicates chlorine.

(c) *Polyvinyl Acetate*.—Place a 0.2-g. sample of elastomer in a test tube with 2 ml. of  $\text{H}_2\text{SO}_4$  (55 per cent) and warm gently. If decomposition occurs, polyvinyl acetate compound is indicated.

(d) *Natural Rubber and Styrene Polymers*:

(1) Asphaltic extenders may interfere with the distinguishing test between styrene type polymers and natural rubber by pyrolysis. If a 0.2-g. sample of elastomer shows an appreciable darkening when shaken with 2 ml. of  $\text{CHCl}_3$ ,

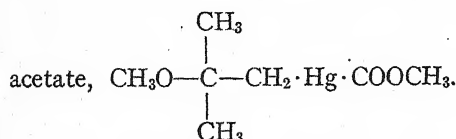
extract a fresh sample with  $\text{CHCl}_3$  in accordance with Section 12 of A.S.T.M. Methods D 297, dry the sample in a vacuum oven for 1 hr. at 70 C., and repeat the pyrolysis test (Section 7).

(2) For further confirmation of the presence of natural rubber proceed as follows: Extract a fresh portion of the sample with acetone in accordance with Section 11 of Methods D 297. Place a few milligrams of the acetone-extracted sample in a small evaporating dish and swell it in a little  $\text{CCl}_4$ . Add a few drops of bromine and let stand for about 2 min. Add about 1 g. of phenol and warm on a steam bath to remove the  $\text{CCl}_4$ . A blue to red-violet color indicates natural rubber. Oil-type reclaimed rubber gives this test but alkali-type reclaim does not. Polyisoprene also gives a positive test.

(3) If doubt still remains as to the presence of styrene polymers, the following test gives positive identification. Boil 1 to 2 g. of dried, acetone-extracted elastomer under reflux with 20 ml. of  $\text{HNO}_3$  (sp. gr. 1.42) for 1 hr. Dilute by pouring into 100 ml. of water and extract with 50-, 25-, and 25-ml. portions of ether. Combine the ether extracts and wash twice with 15 ml. of water, rejecting the washings. Extract the ether solution with three 15-ml. portions of  $\text{NaOH}$  (50 g. per l.) and finally with 20 ml. of water. Discard the ether. Combine the caustic extracts and washing, make just acid with  $\text{HCl}$  (sp. gr. 1.18), and add 20 ml. in excess. Heat on a steam bath and reduce the nitrobenzoic acid by adding 5 g. of granulated zinc. Make the solution alkaline with  $\text{NaOH}$  (200 g. per l.), adding sufficient excess to just dissolve the zinc hydroxide that forms. Extract twice with ether and discard the ether. Make the aqueous solution acid with  $\text{HCl}$  (sp. gr. 1.18), cool to room temperature, and add 2 ml. of 0.5 N  $\text{NaNO}_2$ . Pour this diazotized solution

into an excess of solution of  $\beta$ -naphthol in NaOH (50 g. per l.). A vivid scarlet color indicates styrene in the elastomer.

(c) *Polyisobutylene Rubbers*.—The destructive distillation of polyisobutylene types yields a white difficultly condensable vapor. A light-yellow mobile oil is obtained. An additional test to confirm the presence of polyisobutylene types may be carried out as follows: Place about 1 g. of the acetone-extracted, dried sample in a test tube provided with a stopper and a bent delivery tube passing through a stopper almost to the bottom of a second test tube having a side arm. Cool the second tube with ice. By means of a delivery tube, connect the side arm of the second test tube to a small open test tube containing 0.5 g. of mercuric acetate in 10 to 15 ml. of methanol. Heat the rubber strongly so that it is virtually all decomposed and distilled off. Reject the liquid that collects in the second test tube. Evaporate the methanol from the third tube but do not heat excessively toward the end of the evaporation. Boil the residue with 25 ml. of petroleum ether (boiling point, 40 to 60 C.) and filter from insoluble matter. Evaporate the filtrate to a small volume, chill in ice and scratch the sides of the vessel to induce crystallization. Dry the mercury derivative at 30 to 40 C. and determine the melting point (about 55 C.). For final confirmation, carry out a mixed melting point determination with the mercury derivative made from known polyisobutylene or isobutylene. The derivative is thought to be methoxy-isobutyl-mercuri



(f) *Thioplasts*.—The thioplasts have not been included in this analytical

scheme because of their ease of identification by means of odor and high sulfur content. Additional tests include the pronounced swelling action of acetone (unlike the other elastomers except unvulcanized nitriles) and the extremely rapid reaction at 40 C. with a mixture of equal volumes of  $\text{HNO}_3$  (sp. gr. 1.42) and  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84). The thioplasts will start to decompose in from 5 to 35 sec. Other elastomers normally take a longer time.

## B. QUANTITATIVE ANALYSIS OF ELASTOMERS

### Application

10. To a considerable extent, the quantitative analysis of elastomers present in a compound is possible. Sections 11 to 16 are intended for use in the quantitative determination of elastomers.

### Determination of Chloroprene and Vinyl Chloride Polymers

11. (a) Determine the chlorine content of the sample in accordance with either Paragraph (b) or (c). From this value, calculate the content of chloroprene or vinyl chloride polymer (Note).

NOTE.—The value obtained for chlorine, nitrogen, or sulfur content in accordance with Section 11, 12, or 13, respectively, may be used to estimate the polymer content if the polymer contains one of these elements. Since these polymers may vary widely in composition, the final estimation is not exact unless other factors in the analysis, such as ash analysis, make it evident which type of chlorine-, nitrogen-, or sulfur-containing rubber is present.

(b) *Recommended Method*.—Place 0.2 to 0.5 g. of finely cut sample in an aluminum boat and cover with granular aluminum. Burn the sample in a combustion furnace at 1250 to 1350 C., using a rapid stream of oxygen (0.5 liters per min.). No catalysts are required in the tube, but the sample shall be pushed into the hot part of the tube in steps to prevent too

rapid combustion. Too rapid combustion is evidenced by the appearance of a deposit of black in the absorption train. Absorb the gases in three absorption bottles or tubes, using sintered glass bubbles in the last two bottles. Use hydrazine sulfate (10 g. per l.) in the first bottle and NaOH (100 g. per l.) in the last two bottles. Hydrogen peroxide may be added to the last two bottles if it is thought that free chlorine is going through the first bottle. Continue the oxygen stream for 10 min. after the sample is in the hottest part of the furnace (total combustion time of 15 to 20 min.). Wash the contents of the bottles and bubblers into a flask and acidify with  $\text{HNO}_3$ . If the sample was compounded rubber, it is also necessary to add the contents of the combustion boat which may contain inorganic chlorides. Titrate the chlorides by a convenient method, preferably the Volhard or modified Volhard method, or determine gravimetrically as  $\text{AgCl}$ .

(c) *Alternative Methods*.—Any other combustion method known to give accurate results may be used. The Parr oxygen bomb method of combustion will normally be satisfactory. The Parr peroxide bomb is satisfactory for an estimate of chlorine content but cannot always be relied upon to give complete combustion, particularly if the sample cannot be finely divided.

#### Determination of Nitrile Polymers

12. Determine the nitrogen content of the sample in accordance with Section 24 (b) of A.S.T.M. Methods D 297, except that a 1-g. sample shall be used. From this value, calculate the content of nitrile polymer (see Note, Section 11).

#### Determination of Thioplasts

13. (a) Determine the sulfur content of the sample in accordance with either Paragraph (b) or (c). From this value,

calculate the content of thioplasts (see Note, Section 11).

(b) Proceed in accordance with Section 14 (a) to (d) of A.S.T.M. Methods D 297, up to the point of addition of  $\text{KClO}_3$ . At this point, add sufficient  $\text{HNO}_3$  to insure the presence of 20 ml. of  $\text{HNO}_3$  (sp. gr. 1.42) and add 10 ml. of  $\text{HClO}_4$  (60 to 70 per cent) (Note). Evaporate to heavy fumes of  $\text{HClO}_4$  on a hot plate behind a safety screen. The volume should be 3 to 5 ml. Cool, dilute, and precipitate the  $\text{BaSO}_4$  as described in Section 14(f) of Methods D 297, omitting the addition of  $\text{HCl}$ .

NOTE: *Caution*.—If an excess of  $\text{HNO}_3$  is present and no lumps of organic material such as unreacted polyisobutylene are present when the mixture is evaporated with  $\text{HClO}_4$ , danger of explosion is negligible. However, a safety screen should be used in all cases.

(c) Elastomers containing polyisobutylene cannot be wet-oxidized and the sulfur must be determined by means of a combustion method with oxygen or in a Parr peroxide bomb. If quantitative results are to be obtained in the Parr bomb, the sample shall be finely divided and combustion shall be essentially complete.

#### Determination of Natural Rubber

14. Determine the content of natural rubber in accordance with Sections 48 to 53 of A.S.T.M. Methods D 297.

#### Determination of Styrene-Type Polymer

NOTE.—This method depends upon the oxidation of the polymer, first by nitric acid and then by alkaline permanganate, and isolation and subsequent titration of the ether-soluble acids. These acids are a mixture in which *p*-nitrobenzoic acid predominates. It has been found experimentally that one equivalent of ether-soluble acid is formed for every styrene molecule present in the polymer. It has also been shown that high-styrene polymers (over 30 per cent styrene in the copolymer) fail to give an equivalent amount of acid. Pure polystyrene gives less than 50 per cent yield of acid. The results are at best an estimate (plus or minus 2 per cent

of styrene), and in heavily compounded rubbers or mixtures of elastomers, the error is frequently greater.

15. (a) Weigh 1 g. of sample (2 g. if compounded with filler), and extract with acetone in accordance with Section 11 of A.S.T.M. Methods D 297. Dry the extracted sample at 100 C., and transfer quantitatively into a 100- to 250-ml. flask fitted with a ground-glass joint to a condenser. Add 20 ml. of  $\text{HNO}_3$  (sp. gr. 1.42), attach the condenser, and heat until reaction starts. Remove the heat until the reaction has abated. External cooling may be necessary in some cases. Boil gently for 6 hr. Add 20 ml. of water down the condenser and cool the flask. Add NaOH (200 g. per l.), slowly near the end, until almost exactly neutral to litmus. Add about 1 ml. in excess, but not more than 2 ml. From 40 to 50 ml. of NaOH (200 g. per l.) will be required.

(b) Pour the contents of the flask into a 500-ml. flask and rinse. Add 50 ml. of  $\text{KMnO}_4$  (40 g. per l.) and swirl. Place on a steam bath and swirl from time to time over a period of 5 hr. If the pink color of the permanganate (observed when swirling) fades, add another 10 ml. of  $\text{KMnO}_4$ . Continue this for the entire 5 hr. If more than two additions are necessary, the solution may have been too alkaline and the results may be low. Filter hot through a Büchner funnel and wash the precipitate several times with water.

(c) Transfer the filtrate and washings to a 500-ml. container and cool. Add 3 *N*  $\text{H}_2\text{SO}_4$  until just acid to congo red paper. Add  $\text{NaHSO}_3$  (40 g. per l.) drop by drop to the solution, while stirring, until no pink color remains. Dilute to 250 to 300 ml. if necessary.

(d) Transfer to a separatory funnel and extract with three 100-ml. portions of ether. Combine the ether extracts in the funnel and extract with 10 ml. of

water. Discard the aqueous layer. Extract the ether solution with 25-, 12.5-, and 12.5-ml. portions of 1 *N* NaOH and finally with 15 ml. of water, discarding the ether solution and combining the NaOH extracts.

(e) Transfer the caustic solution to a separatory funnel, acidify with 3 *N*  $\text{H}_2\text{SO}_4$ , and extract with three 50-ml. portions of ether. Extract the combined ether extracts with two 10-ml. portions of water to remove traces of mineral acid. Evaporate the ether in a light flask or beaker, dry by evaporating with a little acetone, and finally dry at 60 C. until the loss in weight is less than 2 mg. after 30-min. heating. Styrene polymers give a crystalline residue.

(f) Dissolve the residue in 25 to 75 ml. of neutral ethyl alcohol (95 per cent) (the amount depending on the color of the solution) and titrate with 0.1 *N* NaOH, using phenolphthalein indicator.

(g) Calculate the percentage of styrene as follows:

$$\text{Styrene, per cent} = \frac{VN \times 0.104}{W} \times 100$$

where:

*V* = milliliters of NaOH solution required for titration,

*N* = normality of the NaOH solution, and

*W* = grams of unextracted sample used.

(h) If there is any question of the identity of the *p*-nitrobenzoic acid, confirm by recrystallizing from acid alcohol-water solution after titration and running a mixed melting point, or by reducing, diazotizing, and coupling as described in Section 9 (d), Item (3). If natural rubber is present, an interference to the extent of about 3 per cent of the rubber present must be subtracted from the styrene content.

#### Determination of Polyisobutylene

NOTE.—This method is moderately successful with mixtures, including compounded or vul-

canized rubbers. If the mixture is compounded or vulcanized, the practical upper limit is about 25 per cent polyisobutylene.

16. Boil 1 g. of finely cut sample with 20 ml. of  $\text{HNO}_3$  (sp. gr. 1.42) under reflux for 30 min. Decant the liquid while hot, wash several times with cold  $\text{HNO}_3$  (sp. gr. 1.42), and then wash with water by decantation. Add  $\text{NH}_4\text{OH}$  (1:2) and warm on the water bath for 30 min. Decant and wash several times with hot water. Dry the insoluble matter at 100 C. and weigh the polyisobutylene so obtained.

#### C. DETERMINATION OF COMPOUNDING INGREDIENTS

##### Procedure

17. Determine compounding ingredients in accordance with the applicable portions of A.S.T.M. Methods D 297, taking into consideration the following points:

(a) *Free Sulfur*.—Free sulfur in polyisobutylene type elastomers may not be quantitatively extracted and determined by either method described in Section 16 of Methods D 297.

(b) *Total Sulfur*.—Total sulfur in polyisobutylene type elastomers cannot be determined by the method described in Section 14 of Methods D 297 because of

insolubility of the elastomer in the reagents. A combustion method must be used.

(c) *Free Carbon*.—When free carbon is determined in accordance with Section 23 of Methods D 297, filtration is materially aided by partial or complete neutralization of the  $\text{HNO}_3$  solution with  $\text{NH}_4\text{OH}$ , particularly with styrene type elastomers. Some synthetic compounds cannot be successfully treated without this neutralization. If the carbon is still too well dispersed to filter after this treatment, the addition of trivalent cations or anions may act as a filter aid in a partially neutralized solution. The carbon in elastomers containing polyisobutylene cannot be determined by the method but may be estimated by retaining, washing, and weighing the washings from the polyisobutylene estimation if the polyisobutylene content is not over 25 per cent. Cellulose from filter paper will slow the filtrations and give high results.

(d) *Cellulose*.—The success of a cellulose determination made in accordance with Section 29 of Methods D 297 will depend on the solubility of the rubber in the solvent.

# *Tentative Method of*

## HEAT AGING OF VULCANIZED NATURAL OR SYNTHETIC RUBBER BY TEST TUBE METHOD<sup>1</sup>



A.S.T.M. Designation: D 865 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

*This method was prepared jointly by the Society of Automotive Engineers and the American Society for Testing Materials*

### Scope

1. This method is intended for use in estimating the relative resistance of vulcanized rubber to high-temperature aging in a controlled and limited quantity of air. No direct correlation between this accelerated test and natural life of rubber is given or implied. Since the rate at which deterioration proceeds during the normal life of rubber varies widely depending on conditions of exposure to heat, light, and air, and on the composition and state of cure of the rubber, this accelerated test is comparative only and must be evaluated against the performance of rubber compounds of which both the natural and accelerated aging characteristics are known.

### Nature of Test

2. (a) This aging test of rubber consists of subjecting test specimens having previously determined physical properties to controlled deteriorating influences

for known periods, after which the physical properties are again measured and the changes noted. In this method the test involves exposure of specimens to air at an elevated temperature and at atmospheric pressure (except as the pressure is increased due to the increased temperature of the unvented test tube). This test is designated as the test tube aging method. It does not include exposure to light.

(b) In this aging test the physical properties used to measure the deterioration of the rubber, in addition to visual and manual inspection, are tensile strength, ultimate elongation, and durometer hardness. Except as otherwise specified in this method, the determination of these properties before and after aging shall be carried out in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412)<sup>3</sup> and the Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676)<sup>3</sup>.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by the Administrative Committee on Standards, February 15, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



## General Methods

3. (a) Except as otherwise specified in this method, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15)<sup>3</sup> shall be complied with and are made a part of this method.

(b) In case of conflict between the provisions of this method and those of detailed specifications or methods of test for a particular material, the latter shall take precedence.

## Apparatus

4. The apparatus for the heat aging shall consist of glass test tubes 38 mm. in diameter and 300 mm. in length heated by immersion in a suitable oil bath so arranged as to comply with the following requirements:

(a) No more than three specimens shall be used in each test tube, and all specimens in a single tube shall be from a single compound.

(b) Provision shall be made for vertical suspension of the specimens as near the bottom of the test tube as possible without touching each other or the sides of the test tube during the test.

(c) The test tubes shall be stoppered with an unvented cork.

(d) The oil bath shall be equipped with thermostatic control which will control the oil temperature within  $\pm 2$  F. of the prescribed temperature, and shall be provided with suitable stirring or circulation to assure uniformity of temperature in all parts of the bath.

(e) A recording thermometer shall be provided to record the actual temperature of the oil bath during the test.

(f) The test tubes shall be immersed in the oil bath so that the space between the surface of the oil and the lip of the test tube does not exceed 2 in.

## Test Specimens

5. (a) Dumbbell-shaped test specimens, prepared as described in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412)<sup>3</sup> shall be considered standard. Their form shall be such that no mechanical, chemical, or heat treatment will be required after aging. If buffing is necessary, it shall be performed prior to the aging cycle.

(b) The dimensions of the test specimens for calculating the physical properties shall be measured prior to starting the aging cycle. Care shall be taken that the material used for marking the gage lines on the specimens is not detrimental to the rubber during aging exposure, causing the specimens to break at the marks prematurely during test. Only specimens of similar dimensions having approximately the same exposed areas may be compared with each other.

## Number of Test Specimens

6. At least three specimens shall be used to determine the average original physical properties of the rubber and also three or more specimens of the same material for each exposure period of the aging test.

## Tests of Unaged Specimens

7. (a) The stress-strain properties or tensile strength and ultimate elongation and the durometer hardness of the original unaged specimens shall be determined within 24 hr. of the start of the aging period. Results on tension test specimens which break outside of the straight reduced section or which are found to be imperfect shall be discarded, and retests shall be made.

(b) When rubber compounds are to be tested for the purpose of determining compliance with a specification, it shall be permissible to determine the original

properties required in Paragraph (a) simultaneously with the determination of the values after the first aging period, even though the elapsed time exceeds 24 hr.

### Procedure

8. (a) The oil bath shall be heated to the temperature selected for the aging test and when the recording thermometer indicates that this temperature is being maintained the stoppered test tubes containing the specimens shall be immersed in the oil as specified in Section 4 (f) and allowed to heat for the desired period. The selected temperature may be in the range of 250 to 300 F. The aging period shall start at the time the specimens are placed in the oil bath. The selection of suitable periods of aging will depend on the rate of deterioration of the particular material being tested. Periods frequently used are 10, 20, 40, 70, and 168 hr. The aging periods used shall be such that the deterioration will not be so great as to prevent determination of the final physical properties. In experimental work, it is desirable to use a range of aging periods, while for routine tests of known materials fewer intervals may be employed.

(b) At the termination of the aging interval, the specimens shall be removed from the test tubes, cooled to room temperature on a flat surface, and allowed to rest not less than 16 nor more than 48 hr. before determination of the physical properties.

(c) When a test follows immediately after another one, care shall be exercised to flush out the test tube with fresh air before proceeding with the second test.

### Physical Tests of the Aged Specimens

9. The stress-strain properties or tensile strength and ultimate elongation and durometer hardness of the specimens aged for different periods shall be deter-

mined as the intervals terminate in the progress of aging, disregarding the fact that more specimens may still be aging. In determining the stress-strain properties after aging, the final values shall be the average of results from at least two specimens, including that one giving the highest value for ultimate tensile strength and any which check that value within 15 per cent. Results of tests of all other specimens shall be discarded. In the event that discarding of aged specimens, both from faulty breaks and failure to check, does not leave two satisfactory determinations, additional specimens shall be aged and tested until two or more check results from properly broken specimens are obtained.

### Calculations and Report

10. (a) The results of the aging test on durometer hardness shall be expressed in terms of the numerical change in indentation reading. The results obtained on the other physical properties (tensile strength, ultimate elongation or tensile stress) shall be expressed as a percentage change based on the original values, increases being indicated as positive and decreases as negative.

(b) The report shall include the following:

- (1) The results calculated in accordance with Paragraph (a),
- (2) All observed and recorded data on which the calculations are based,
- (3) Type of aging test,
- (4) The aging interval,
- (5) The aging temperature,
- (6) The duration, temperature, and date of vulcanization of the rubber, if known,
- (7) Dates of original and final determinations of physical properties, and
- (8) Dimensions of test specimens.

# *Tentative Method of Test for*

## RESISTANCE TO ACCELERATED LIGHT AGING OF RUBBER COMPOUNDS<sup>1</sup>



A.S.T.M. Designation: D 750 - 43 T

ISSUED, 1943.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method is intended for use as an accelerated test applicable for estimating the comparative resistance of soft vulcanized rubber compounds to deterioration when exposed to light having a frequency range approximating that of sunlight, but a greater intensity in the ultraviolet range than sunlight. This method is not intended to cover the testing of materials ordinarily classed as hard or semihard rubber.

### Nature of Test

2. This test consists of continuously or intermittently exposing rubber specimens held in a jig or holder under a specified strain to the effect of light having essentially the same wave lengths as are found in natural sunlight but with increased intensity in the ultraviolet range. The primary criterion used in estimating resistance to light aging is the percentage decrease in tensile

strength and in elongation at break. A supplementary criterion for estimating resistance to light aging is the observed extent of surface crazing and cracking.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Weathering Unit.*<sup>3</sup>—The weathering unit shall include means for measuring and controlling the following:

Current,  
Voltage,  
Temperature of air and water, and  
Running time.

The materials of construction shall be of such character as not to react with the test specimens during the test.

(b) *Light Source.*—A carbon-arc light source. The carbons shall be of such composition and shall be operated under such conditions that the quality of the

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, November 20, 1943.

<sup>3</sup> Any accelerated weathering unit which meets the requirements prescribed in Section 3 may be used. The following types of weathering units have been found suitable:

National Accelerated Weathering Unit X-1A produced by the National Carbon Co.  
Twin Arc Weatherometer, type DL-TS produced by the Atlas Electric Devices Co.

spectrum as it strikes the test specimens shall approximate the spectrum of the sun.

(c) *Specimen Supports*.—Supports for mounting the specimens vertically while they are rotated about the carbon arc to provide uniform distribution of light. If the specimens are mounted both above and below the horizontal center line of the light source, they may be mounted at an angle with the vertical not greater than 30 deg., so that the light from the arc has a more nearly normal incidence upon the specimens. When specimens are mounted angularly, the angle that the specimens make with the vertical shall be reported. Specimens mounted above and below the center line of the light source shall be transposed periodically to provide uniform distribution of the light over the face of the panel. Arc vapors or products shall not be allowed to contaminate the specimens.

(d) *Water Sprays*.—Water sprays whereby clean water, such as drinking water, may be forced on the specimens to simulate the washing action of rain, to provide moisture for causing alternate expansion and contraction due to swelling and drying out, and to introduce thermal shock and sweating. Recirculation of or immersion in the spray water shall not be permitted.

(e) *Thermometer*.—A thermometer for determining the temperature of the air at the position of the specimen in the drum. The bulb of the thermometer shall be shielded by a cylinder of bright metal foil 2 in. in diameter and 2 in. in length. The thermometer bulb shall be located centrally in the foil cylinder.

#### Calibration of Radiation

4. The light source shall be calibrated at frequent intervals in accordance with the Tentative Method of Calibrating a Light Source Used for Accelerating the

Deterioration of Rubber (A.S.T.M. Designation: D 749).<sup>4</sup>

#### Test Specimens

5. Test specimens shall be rectangular strips 3 in. in width by 6 in. in length prepared especially for this test or cut from the material which is to be evaluated. The specimens should have a maximum thickness of 0.030 in. and a minimum thickness of 0.025 in. Specimens cut from rubber products may be buffed. Duplicate specimens should be exposed whenever possible.

#### Precautions

6. In the operation of the weathering unit, care should be taken to see that the machine at all times is being operated under constant voltage at the recommended arc amperage, that the proper carbons are used, that the globes and filters are cleaned at frequent intervals, and that the temperatures of the air and the water are regulated within the tolerances specified.

#### Procedure

7. (a) The test specimens shall be firmly fastened in the jig or holder and elongated if desired but will usually be exposed at no elongation. The specimen shall then be exposed to the light source until a dosage or total exposure equivalent to the decomposition of the desired number of milligrams of anhydrous oxalic acid as determined by the calibration procedure (Section 4) has been reached. The temperature of the air surrounding the specimen shall be maintained between 35 and 45 C. The average temperature of the air during the test shall be recorded. The water flow in cubic centimeters per minute through the sprays during each test shall be measured and recorded. The temperature of the water at the spray shall be main-

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

tained between 10 and 25 C. and the approximate average water temperature during the test shall be recorded. The chlorine content of the water shall be determined and reported. At the conclusion of the exposure period, the specimen shall be removed from the testing unit and examined for degree and number of cracks and for evidence of crazing.

(b) After examination of the exposed specimen, three tension test specimens shall be prepared from it in accordance with the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15).<sup>4</sup> The tensile strength and ultimate elongation of these exposed specimens shall be determined in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412).<sup>4</sup> The tensile strength and elongation of duplicate unexposed specimens of the same material shall also be determined for purpose of comparison at the same time that the exposed specimens are tested.

(c) A test for the presence in the testing unit of ozone adjacent to the exposed specimens shall be made by exposing a small filter paper previously dipped in starch - potassium iodide solution. Ozone, if present, will cause the test paper to turn blue. The length of time required for the formation of a deep blue coloration may be taken as an indication of the quantity of ozone present. The test paper shall be shielded from the light source. An alternative test for the presence of ozone may be made as follows: Select a 1- by 3-in. sample of the control stock in the material being exposed and stretch to an elongation of 25 per cent. Place it in the accelerated aging unit at the position of the irradiated specimens but shielded from the direct rays of the arc. If avail-

able, expose a 1 by 3 by 0.1-in. sample of natural rubber or GR-S black tire tread stock similarly and simultaneously. The time of exposure necessary to form the first visible cracks shall be reported to indicate the ozone concentration. The number and type of cracks shall also be reported.

### Report

8. The report shall include the following:

(1) Description and dimensions of specimens,

(2) The number of cracks and degree of crazing or cracking of the specimens,

(3) The intensity of the light source expressed as the number of milligrams of anhydrous oxalic acid decomposed per square decimeter per minute,

(4) The total exposure expressed in equivalents of the number of milligrams of anhydrous oxalic acid decomposed at the rate determined by the average intensity of exposure multiplied by the total length of time of exposure,

(5) The percentage of elongation during exposure, if any,

(6) Tensile strength in pounds per square inch before and after exposure,

(7) Ultimate elongation at break before and after exposure,

(8) The percentage loss in tensile strength as a result of exposure,

(9) The percentage loss in elongation at break as a result of exposure,

(10) Indication of ozone,

(11) Average temperature at position of test specimens during exposure to the light source,

(12) Temperature and rate of flow of water in cubic centimeter per minute in the water sprays,

(13) Chlorine content of the water, and

(14) Name and type of accelerated aging unit used, and the cycle and operating conditions used.

## *Tentative Method of*

### CALIBRATING A LIGHT SOURCE USED FOR ACCELERATING THE DETERIORATION OF RUBBER<sup>1</sup>



A.S.T.M. Designation: D 749 - 43 T

ISSUED, 1943.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. (a) This method is intended primarily for use in measuring the intensity of radiation from the light source used in connection with the Tentative Method of Test for Resistance to Accelerated Light Aging of Rubber Compounds (A.S.T.M. Designation: D 750).<sup>3</sup> It may also be used for measuring and integrating the intensity or total exposure from other light sources which are intermittent or variable in intensity, such as sunlight. It may also be used for comparing light sources with regard to ultraviolet intensity.

(b) It must be emphasized that calibrations made as described in this method are essentially a measure of the ultraviolet dosage of a given source of light. Such calibrations do not permit the comparison of light sources differing in spectral distribution unless further in-

formation is available. However, this method will determine the constancy of a specific source, and may be used to compare the intensities of similar light sources.

#### Nature of Test

2. This method is based on the fact that oxalic acid in the presence of uranyl sulfate in aqueous solution is photochemically decomposed by light, essentially at a rate in direct proportion to the intensity and total radiation of the light source to which the solution is exposed over the wave length range used for the test. The test method involves the exposure of a standardized oxalic acid - uranyl sulfate solution in a quartz cell to the radiation to be evaluated, followed by a determination of the quantity of oxalic acid decomposed during a measured time period of exposure.

#### Apparatus

3. The apparatus shall consist of the following:

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, November 20, 1943.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(a) *Actinometer Cell*.—The actinometer cell shall be cylindrical in form with circular plane-polished quartz windows 1.5 mm. in thickness. A side tube 6 mm. in diameter and 1 cm. in length and suitable for filling and emptying the cell shall be fused to the cylinder wall. The dimensions of the completed cell shall be 5 cm. in internal diameter and 1 cm. in internal length. The cell windows may be either fused or cemented to the rest of the cell. Where the windows are cemented, one plane-polished window and the cylindrical body of the cell may be made of pyrex, Corex D, or other clear glass, provided that the other window of the cell which shall face toward the light during actinometric measurements shall be of plane-polished quartz 1.5 mm. in thickness. In cemented cells the cement used shall not dissolve in nor react with the actinometer solution.

(b) *Cell Enclosure*.—The actinometer cell used shall be enclosed in a lightproof box coated on the inside with an highly absorbent black paint. The cell shall be totally covered in the enclosure, except for one face which shall be exposed through an opening in the enclosure conforming to the full circular cross-section of the solution in the cell.

#### Reagents

4. (a) *Potassium Permanganate Evaluating Solution (3.16 g. per l.)*.—Prepare an evaluating solution containing 3.16 g. of  $\text{KMnO}_4$  per liter. Dissolve the  $\text{KMnO}_4$  in distilled water, store the solution in the dark for at least one week, then filter through a fibrous glass filter cloth  $\frac{1}{8}$  in. in thickness, and place the solution in a blackened glass-stoppered bottle or automatic dispensing burette. Standardize against sodium oxalate (Note) in accordance with the procedure of Kolthoff and Furman.<sup>4</sup>

(b) *Actinometer Solution*.—Dissolve 6.30 g. of c.p., reagent grade oxalic acid crystals  $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$  and 4.20 g. of c.p. reagent grade uranyl sulfate ( $\text{UO}_2\text{SO}_4 \cdot 3\text{H}_2\text{O}$ ) in distilled water and dilute to 1 liter. (This solution will be 0.1 N with respect to oxalic acid.) Immediately after preparation, transfer the solution to a glass-stoppered bottle or to the reservoir of an automatic dispensing burette. The bottle, or reservoir and glass tubes of the burette, should be protected from light by a heavy coating of black enamel. Standardize (Note) as follows: Transfer an accurately measured 50-ml. portion of the solution to a 200-ml. tall-form beaker, dilute with 20 to 25 ml. of distilled water, and acidify with 5 ml. of  $\text{H}_2\text{SO}_4$  (1:3). Cover with a suitable crystallizing dish placed in an inverted position on the beaker, and heat the solution to 95 C. in a lightproof water bath. When the solution reaches 95 C., transfer the beaker to an open glass water bath maintained at 95 C. and resting on a flat, white-glass base. Titrate the hot solution by adding the  $\text{KMnO}_4$  evaluating solution (3.16 g. per l.) from a burette while stirring constantly. Add the first few drops of evaluating solution at intervals of several seconds. The end point is indicated by the change to orange color which should persist for 30 sec. while stirring.

NOTE.—Whenever a fresh solution is added to either burette, or if either solution should stand for 60 days or more, restandardize the solutions.

#### Procedure

5. (a) A suitable quantity of actinometer solution shall be measured accurately into the actinometer cell from the dispensing burette holding the solution. The cell shall always be filled with the same amount of actinometer solution. The quantity used shall be such that the solution does not extend excessively into

<sup>4</sup>I. M. Kolthoff and N. H. Furman, "Volumetric Analysis," Vol. II, pp. 282-285, John Wiley and Sons, Inc., New York, N. Y.



the neck of the cell but is just sufficient to prevent the formation of an air bubble when the cell is inclined at an angle of 45 deg. Care shall be taken to exclude all stray light from the solution while transporting it to and from the light source under test by enclosing the cell in a suitable lightproof box. The cell shall be mounted on a rubber specimen exposure rack in the testing unit and exposed for a suitable period of time to the light source under normal operating conditions of the testing unit. The time of exposure of the actinometer solution to the light source shall not be in excess of that required for the decomposition of 30 per cent of the oxalic acid present.

(b) At the end of the period of exposure, the actinometer solution shall be emptied into a 200-ml. tall-form beaker, the cell rinsed with 25 ml. of distilled water and the solution and wash water acidified with 5 ml. of dilute  $\text{H}_2\text{SO}_4$  (1:3) per 50 ml. of original undiluted actinometer solution. The beaker shall then be covered, heated to 95 C. and the solution titrated with the  $\text{KMnO}_4$  evaluating solution exactly as in the standardization procedure as described in Section 4 (b).

(c) *Blank.*—A blank test shall be made under the same conditions except that the actinometer solution shall not be exposed to the light source and the decomposition values adjusted accordingly.

### Calculations

6. (a) The light intensity shall be calculated as follows:

$$I = \frac{(A - B) \times C}{D \times E}$$

where:

$I$  = intensity of radiation expressed in milligrams of oxalic acid decomposed per square decimeter per minute,

$A$  = milliliters of evaluating solution equivalent to the number of milliliters of unexposed actinometer solution used in the test,

$B$  = milliliters of evaluating solution required for titration of the actinometer solution exposed in the test,

$C$  = milligrams of oxalic acid equivalent to 1 ml. of evaluating solution,

$D$  = time of exposure in minutes, and

$E$  = exposed area in square decimeters of solution in cell.

NOTE: *Example.*—A typical calculation for evaluating light intensity is as follows:

Area of solution in cell exposed	0.16 sq. dm.
Time of exposure	100 min.
Volume of actinometer solution used in test	50 ml.
Volume of evaluating solution required by 50 ml. of unexposed actinometer solution	50 ml.
Volume of evaluating solution required by exposed actinometer solution	35 ml.
Decomposition expressed as milliliters of evaluating solution (50-35)	15 ml.
Milligrams of oxalic acid ( $\text{H}_2\text{C}_2\text{O}_4$ ) equivalent of 1 ml. of evaluating solution (0.1 N)	4.5 mg.
Total quantity of oxalic acid decomposed (15 × 4.5)	67.5 mg.
Intensity = $\frac{67.5}{100 \times 0.16}$	= 4.218 mg. of oxalic acid per min per sq. dm.

(b) Where exposure to the light source is intermittent or of variable intensity, the total or integrated exposure or "dosage" only is determined and shall be reported in terms of milligrams of oxalic acid decomposed per square decimeter during the time of exposure.

*Tentative Method of Test for*  
PHYSICAL STATE OF CURE OF VULCANIZED RUBBER  
(T-50 TEST)<sup>1</sup>



A.S.T.M. Designation: D 599 - 40 T

ISSUED, 1940.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. This method of test, known as the "T-50 test," is designed as a means of determining the extent or physical state of cure of a rubber compound by measuring the temperature at which it recovers its elasticity when it is stretched at room temperature, frozen at a sufficiently low temperature to cause it to lose its elastic properties, and then gradually warmed. When the test is carried out under properly controlled conditions, the "T-50 value" shows a good correlation with combined sulfur in a stock of given composition, and a qualitative correlation with combined sulfur in stocks of varying compositions. Its advantages over a combined sulfur determination are its greater speed and simplicity, and its applicability in certain cases where sulfur determinations are difficult or impossible. Its use is limited to rubber compounds with a

relatively high elongation at break (generally 300 per cent or over). It is mainly useful in controlling and checking uniformity of cure in production, and as a tool in experimental work where knowledge of the state of cure is important. It is not suitable for use in purchase specifications.

#### Nature of Test

2. A test specimen 1 to 2 in. in length and having a uniform cross-section, is immersed in a liquid bath at 20 C. stretched to an elongation of 75 per cent or more of the breaking elongation, and held for 5 min. It is then plunged into a bath of acetone at -50 to -70 C., held until cool, and released. The rubber will show practically no elastic retraction. The temperature is then raised slowly and the sample is found to contract, taking a definite length at a given temperature. The temperature on the Centigrade scale at which the elongation of the sample becomes equal to one half the initial elongation is the "T-50 value."

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, September 26, 1940.

## Types of Apparatus

3. (a) There are two types of apparatus in general use, designated types *A* and *B*, either of which give satisfactory results when properly operated. Both types perform the same functions and shall conform to the requirements prescribed in the following Paragraphs (b) to (e). The two types of apparatus are described in further detail in Sections 5 and 7.

(b) *Testing Bath and Fittings*.—A container well insulated against heat transfer is required to hold the cold acetone bath. The container shall be fitted with a support to hold the specimen racks, an immersion heater equipped with an on-and-off switch, a thermometer graduated from  $-70$  to  $+30$  C. in 1 C. graduations, and means for circulating the bath liquid in order to maintain a uniform temperature throughout the bath.

(c) *Refrigerating System*.—The refrigerating system shall be capable of rapidly cooling the acetone bath to a temperature of  $-50$  to  $-65$  C. (Note 1). A suitable system consists of a copper coil packed with a mixture of dry ice and acetone in which the acetone for the testing bath is cooled by contact.

(d) *Conditioning Bath*.—The conditioning bath shall consist of a container (preferably with heat insulation) of sufficient capacity to accommodate the specimen racks, and to keep the test specimens completely immersed. It shall be filled with water at a temperature of  $20 \pm 1$  C. (this temperature may be hand regulated), except for testing latex compounds when acetone shall be used instead of water.

(e) *Specimen Rack*.—The specimen rack shall consist essentially of a metal support equipped with a fixed clamp at one end and a movable clamp at the other end for holding the test specimens.

The clamps measure off a predetermined length of the specimen, which is then elongated to the desired amount by moving the movable clamp. Arrangements shall be made for fastening the movable clamp at positions corresponding to the desired elongation of the specimens, and to one half of this elongation.

## Test Specimens

4. The test specimens for use with clamps of the type illustrated in Fig. 2 may be prepared by dieing out with a die of the design shown in Fig. 1. The

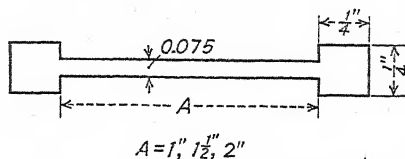


FIG. 1.—Die for Preparing Test Specimens.

choice of die length is governed by the elongation required and the limitations of the specimen racks. For most work a  $1\frac{1}{2}$ -in. die is suitable. Any other method of obtaining test specimens of uniform cross-section is satisfactory, provided that a suitable clamp is used on the rack.

## Method A

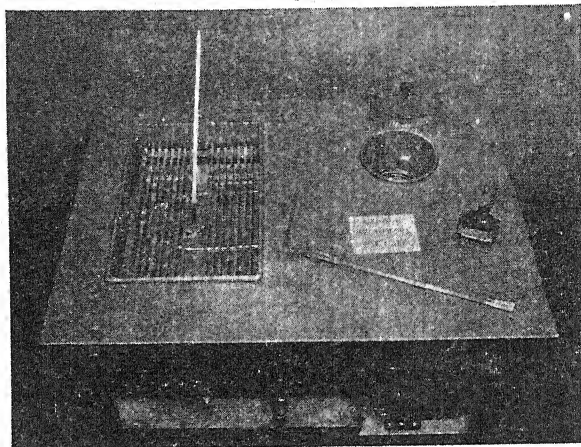
### Type A Apparatus<sup>3</sup>

5. (a) Type A apparatus, illustrated in Fig. 2 (a) and (b), consists of the parts described in the following Paragraphs (b) to (e). This type of apparatus is suitable for use when samples are available in the form of sheets 0.02 to 0.1 in. in thickness and of sufficient size to permit the dieing out of test specimens with the standard die (Fig. 1). Due to the use of individual racks in type A apparatus, it is advantageous for use

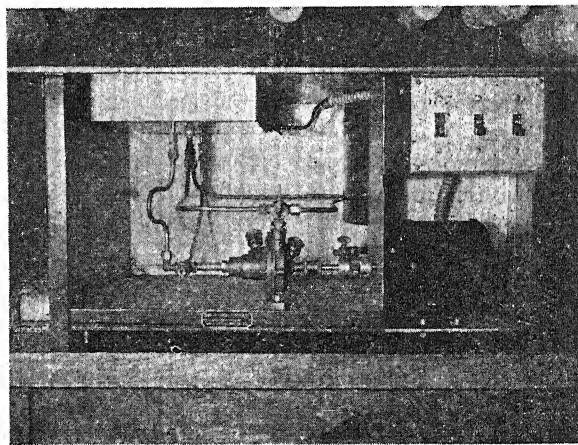
<sup>3</sup> Type A apparatus is described in a paper by W. F. Tuley on "Rubber Compounding with the T-50 Test," *India Rubber World*, Vol. 97, No. 1, October 1, 1937, p. 39.

when specimens requiring different degrees of elongation are to be tested. The fire hazard is somewhat greater with type A apparatus.

Circulation is maintained by means of a pump, which takes acetone out from two outlets at the ends of the testing bath, and forces it in at the center of



(a) Top View.



(b) Front View.

FIG. 2.—Type A Apparatus.

(b) *Testing Bath*.—The testing bath consists of a shallow insulated container open at the top, equipped with a support to hold the specimen racks in a horizontal position, and a support to hold the thermometer. An electric immersion heater equipped with an on-and-off switch is located in the bath.

the bath. A valve is provided to bypass the circulating acetone through the cooling coil during the cooling cycle.

(c) *Refrigerating System*.—The refrigerating system consists of a copper coil enclosed in an insulated tank. The tank is packed with a mixture of dry ice and acetone, and the acetone for the

testing bath is cooled by circulating it through the coil (Note 3).

(d) *Conditioning Bath*.—A conditioning bath is not provided; however, any container of a size suitable for holding the specimen racks when immersed may be used.

(e) *Specimen Racks*.—Sixteen racks, each holding one test specimen, are provided as shown in Fig. 2 (a). A fixed clamp at one end and a movable clamp hold the ends of the specimen, which have tabs on the ends like those produced by the die shown in Fig. 1. The movable clamp is held in a position corresponding to any desired elongation of the specimen by means of an adjustable stop; a latch is used to fasten the clamp to this stop, so that it is easily disengaged when desired. A second adjustable stop holds the movable clamp at a position corresponding to one half of the original elongation.

#### Procedure

6. (a) The apparatus shall be prepared for use by packing the cooling chamber carefully with dry ice. The system shall then be carefully filled with acetone (Caution—acetone is inflammable) by adding the acetone directly to the open pan. The circulating pump shall be started and the valves set so that the acetone is circulated through the refrigerating coils and additional acetone added until the entire system is filled to a point about  $\frac{1}{2}$  in. above the specimens in the racks. The acetone may be precooled before being added to the system, but should never be cooled by direct contact with dry ice.

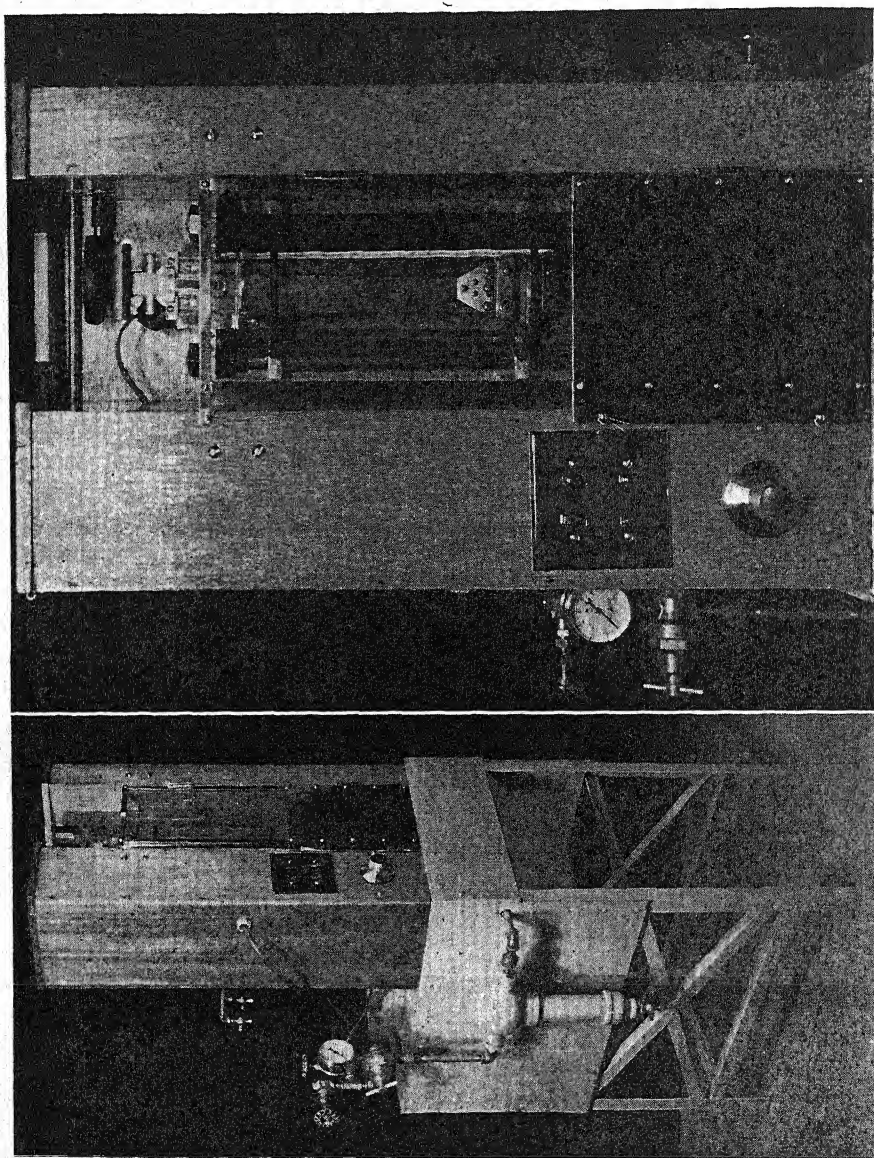
(b) The thermometer shall be set up with the bulb immersed in the center of the bath. To prevent direct contact of the bulb with metal it should be protected with a thin rubber sac or rest on a rubber pad. When the tempera-

ture of the bath reaches  $-50^{\circ}\text{C}$ . (Note 1) or below, the apparatus is ready for the specimens.

(c) The specimen racks shall be prepared by adjusting the latch stop to the predetermined elongation, which is approximately 75 per cent (Note 2) of the ultimate elongation of the material under test, and setting the sliding stop between the grips so that the movable grip contacts this stop at one half of the racked elongation.

(d) The test specimens, conforming to Fig. 1, shall be set up in the racks and the jaws tightened. The racks shall then be immersed in the conditioning bath and the specimens immediately racked until the latch engages the stop which has been preset at the specified elongation. The racked specimens, which shall be fully immersed, shall be left in the conditioning bath for 5 to 10 min., removed, rinsed in an acetone bath at  $15^{\circ}\text{C}$ ., and placed in the cold acetone bath. Under no condition should the specimens be allowed to remain in the conditioning bath for more than 10 min. After the racks have been placed in the cold acetone bath, the temperature of the bath shall again be lowered (there will be a rise in temperature due to the mass which has been added) and the specimens allowed to remain racked in the bath for 1 min. after the bath temperature has again reached  $-50^{\circ}\text{C}$ .

(e) The test specimens shall be released (there will be no immediate evidence of retraction) and the valve controlling the circulation of the bath through the refrigerating system closed. The pump should remain in operation to insure circulation and uniform temperature throughout the bath. The immersion heater shall be turned on and manually operated to the extent that the temperature rises at a rate not ex-



(a) General View.

(b) Front View.  
FIG. 3.—Type B Apparatus.



ceeding 1 C. per min. Practically, the accuracy and reproducibility of the test is not impaired if the temperature of the bath is raised at a rate of 5 to 10 C. per min., provided that the rate of 1 C. per min. is maintained at the T-50 point.

(f) *T-50 Value*.—The temperature at which the specimen just becomes taut shall be noted and recorded as the T-50 value. When working with specimens of pure gum or latex, or specimens of small cross-section, it is desirable to set the specimen manually to the T-50 point immediately after release to prevent error due to drag.

### Method B

#### Type B Apparatus<sup>4</sup>

7. (a) Type B apparatus consists of the parts described in the following Paragraphs (b) to (e). A general view of type B apparatus and a front view showing the testing bath and equipment therein appears in Fig. 3. A diagrammatic sketch illustrating the operation of this apparatus is shown in Fig. 4. Type B apparatus is especially suitable for testing specimens of light-weight material such as rubber thread or thin sheets due to the arrangement of the specimen racks in which up to 20 specimens may be tested at one time. With this apparatus, it is necessary to give all specimens the same elongation in an individual test.

(b) *Testing Bath*.—The testing bath consists of a Dewar tube mounted behind a protective glass window as shown in Fig. 3 (b). This figure also illustrates the mounting of the heater, the air-driven stirrer, the thermometer, and the specimen rack in the testing bath.

(c) *Refrigerating System*.—The refrigerating system is located at the rear and below the table top. It consists of an insulated container for the cooling mixture of dry ice and acetone with a copper coil inside the container, and a reservoir partially surrounding the outside of the container (Fig. 4). The cooling coil is connected at one end to the reservoir and at the other end through the valve 3 to the testing bath. The top of the reservoir is connected through the valve 1 to a supply of com-

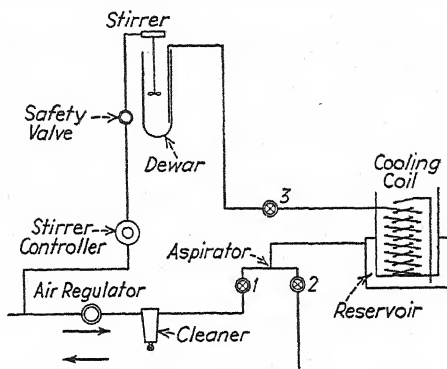


FIG. 4.—Diagram Showing Operation of Type B Apparatus.

pressed air which can be utilized either to force acetone into the testing bath by air pressure (valve 1 open, valve 2 closed) or to siphon acetone out of the testing bath into the reservoir by suction (valves 1 and 2 both open):

(d) *Conditioning Bath*.—Two conditioning baths, one for acetone and one for water are provided in the upper left-hand portion of the cabinet (Note 3). The drain cocks are visible in Fig. 3 (a).

(e) *Specimen Rack*.—A type of rack suitable for use with the type B apparatus is illustrated in Fig. 5. It consists of a brass channel frame with the fixed clamp mounted at the lower end. The movable clamp slides along the frame and is moved by means of a strip

<sup>4</sup> Type B apparatus is described in a paper by W. A. Gibbons, R. H. Gerke, and H. C. Tingey on "The T-50 Test for State of Cure," *Industrial and Engineering Chemistry Analytical Edition*, Vol. 5, No. 4, July 15, 1933, p. 279.



of brass extending beyond the top of the rack. The movable clamp is held in the desired positions by means of slots in the sides of the brass strip which engage a fixture at the top of the rack. A spring keeps the slots engaged with the fixture until it is desired to release the movable clamp. This rack is capable of holding up to 20 specimens at a time.

then be rinsed in an acetone bath at about 15 C. and transferred to the cold acetone bath. (If an acetone conditioning bath is used, rinsing is not necessary). The acetone shall be alternately transferred to the cooling reservoir and back to the testing bath until the temperature of the testing bath has reached at least  $-50^{\circ}\text{C}$ . (Note 1). The air stirrer shall be run at a sufficient speed to maintain a

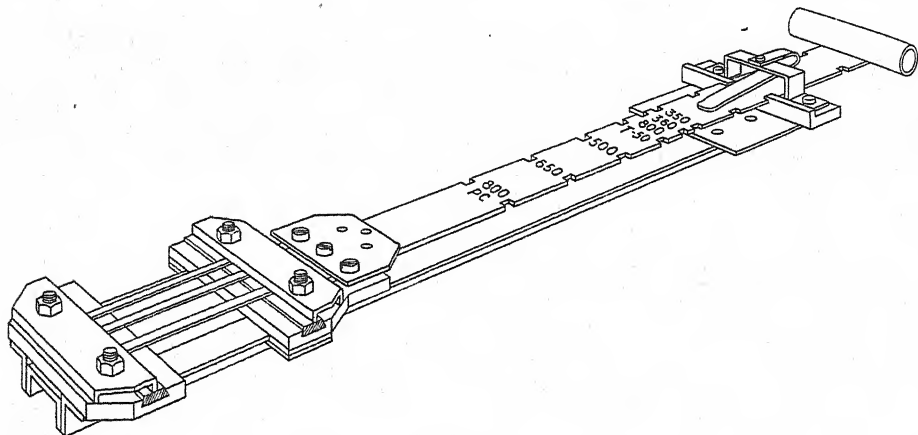


FIG. 5.—Specimen Rack for Type B Apparatus.

### Procedure

8. (a) The cooling container shall be filled with a mixture of dry ice and acetone, and the testing bath, cooling coil, and cooling reservoir filled with acetone to the desired level. The acetone in the testing bath shall be brought to a temperature of  $-50^{\circ}\text{C}$ . or lower by alternately transferring it to the cooling reservoir and back to the bath.

(b) The test specimens shall be clamped in the rack and then placed in the conditioning bath at  $20^{\circ}\text{C}$ . After the specimens in the rack have reached the temperature of the conditioning bath they shall be stretched to the specified elongation (Note 2) and allowed to stand for 5 min. If water is used in the conditioning bath the specimens shall

uniform temperature in the testing bath. The specimens shall be allowed to remain racked in the bath for at least 1 min. after the bath has reached the desired temperature.

(c) The specimens shall be released, the upper clamp moved to a position corresponding to one half of the original elongation of the specimens, and the immersion heater turned on. By opening and closing the heater switch a rate of temperature rise not exceeding  $1^{\circ}\text{C}$ . per min. may be maintained. (During the early stages of heating, rates of temperature rise of 5 to  $10^{\circ}\text{C}$ . are permissible, provided the rate is slowed down to  $1^{\circ}\text{C}$ . per min. by the time the temperature is  $5^{\circ}\text{C}$ . below the T-50 point

of the specimen being tested.) During the heating, the specimens, which on release show practically no elastic recovery, will gradually contract.

(d) *T-50 Value*.—The temperature at which a specimen becomes just taut between the clamps shall be taken as the T-50 value for the specimen.

## Report

9. The report should include the following:

- (1) The T-50 value,
- (2) The elongation used in the test, and
- (3) The ultimate elongation of the specimens.

## EXPLANATORY NOTES

NOTE 1: *Initial Bath Temperature*.—An initial bath temperature of  $-50^{\circ}\text{C}$ . is adequate for practically all ordinary work. In testing specimens in which the T-50 values are expected to be under  $-30^{\circ}\text{C}$ . the initial bath temperature should be dropped to  $-65^{\circ}\text{C}$ .

NOTE 2: *Effect of Elongation*.—As the elongation to which the specimen is stretched before freezing is increased, the T-50 value becomes higher, approaching constancy as the ultimate elongation is approached. With accelerated "pure gum" stocks, little or no change occurs above 500 per cent elongation. With accelerated tire tread stocks (40 parts of carbon black per 100 parts of rubber), elongations of 350 per cent and higher usually give constant results. With unaccelerated stocks, the T-50

value increases appreciably even above 650 per cent elongation. In any case, stretching to 75 per cent of the breaking elongation will give reproducible results.

NOTE 3: *Precautions*.—It is recommended that the apparatus be drained of acetone, if left standing unused for any period of time. The acetone should be filtered before replacement and special pains taken to free it of water which is troublesome in such a low-temperature system. It is necessary to replace the acetone from time to time due to discoloration; it can, however, be redistilled and re-used if adequate equipment for the distillation is available. *Caution*, never lose sight of the fact that acetone is inflammable and should be carefully handled.

# *Tentative Method of Test for*

## INDENTATION OF RUBBER BY MEANS OF THE DUROMETER<sup>1</sup>



A.S.T.M. Designation: D 676 - 46 T

ISSUED, 1942; REVISED, 1944, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers the procedure for determining the indentation of rubber by means of the durometer. The method is considered a convenient means for obtaining an indication of the approximate hardness of rubber articles. It is not suitable for general use in purchasing specifications, except when the values specified are accompanied by adequate tolerances.

### Apparatus

2. (a) *Indenter Point*.—The indenter point shall be made of hardened steel and shall conform to the shape and size shown in Fig. 1.

(b) *Calibrated Spring*.—The spring in the durometer shall be such that when it is calibrated in accordance with the procedure described in the paper on "The Standardization of Durometers," by Lewis Larrick<sup>3</sup> the calibration curve

shall conform to a straight line, one end of which is at zero dial reading and 50-g. load and the other end at 100 dial reading and 850-g. load, as shown in Fig. 2. A variation of plus or minus 4 g. from the straight line shall be permitted at any or all points.

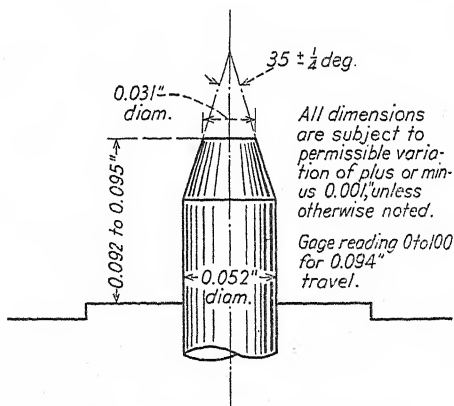


FIG. 1.—Indenter Point for Durometer.

### Test Specimens

3. Test specimens shall be at least 0.25 in. in thickness. Thinner samples may be piled up to obtain this thickness,

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> *Proceedings*, Am. Soc. Testing Mats., Vol. 40, p. 1239 (1940).

but hardness determinations made on such specimens shall be considered only indicative and not definite. No hardness determination shall be made within 0.5 in. of the edge of the specimen and under no circumstances shall hardness

(1) The durometer and the specimen shall be conditioned in air or in a mixture of air and carbon dioxide at the prescribed temperature for at least 30 min. before test.

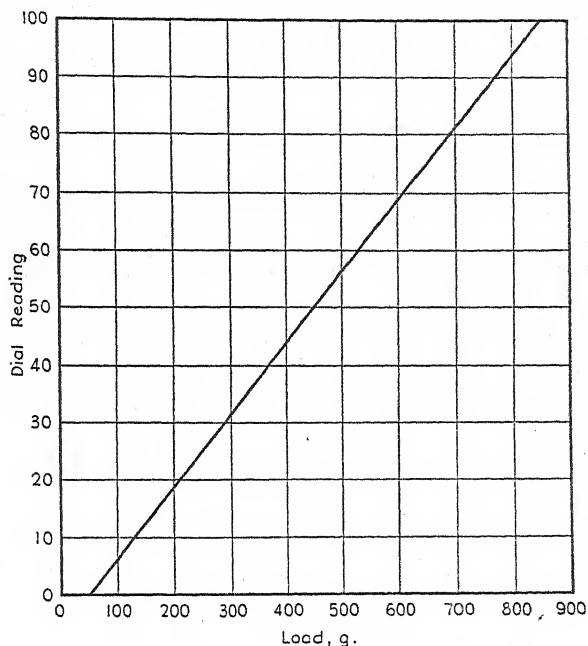


FIG. 2.—Calibration Curve for Durometer Spring.

determinations be considered practical unless the entire width of the bearing plate is resting on the specimen. A suitable hardness determination cannot be made on a rounded surface or on one that is narrower than the bearing plate.

#### Temperature of Test

4. (a) *Normal Temperature of Test.*—The durometer test shall normally be conducted with both the instrument and specimen stabilized at room temperature, which shall be between 70 and 90 F. (21 and 32 C.).

(b) *Use at Temperatures Other than Normal.*—When tests are made at temperatures other than normal, the following conditions shall be observed:

(2) Several determinations shall be made by manual or suitable mechanical manipulation.

(3) Whenever, for any reason, it becomes necessary to remove the durometer from a chamber at a temperature below room temperature, the durometer shall be placed in a suitable dessicator immediately upon such removal and allowed to remain there until its temperature is above the dew point of the air in the room.

(4) A durometer used for testing at other than normal temperatures shall not be used for subsequent determinations until it has definitely attained room temperature.

(5) All readings taken at other than normal temperature shall be recorded with a notation clearly stating the actual temperature of test.

#### Procedure

5. (a) *Method of Application of Durometer to the Specimen.*—The durometer shall be grasped between the thumb and the third and fourth finger, resting the index finger on top of the instrument. The durometer shall then be placed on the test specimen while keeping the base of the instrument parallel to the surface of the specimen. This method of application allows the seating of the plate around the indenter point flush with the test surface with a minimum amount of rocking.

(b) *Pressure Application.*—The test pressure applied shall be just sufficient to insure firm contact of the pressure plate with the test specimen.

(c) *Time of Reading.*—The durometer indentation reading shall normally be taken immediately after application of the instrument so as to obtain the maximum numerical value. In the event that this immediate reading is not suitable for the type of work being done as in research or for investigations of flow properties or the like, it is permissible to use readings taken 15 sec. after application of the instrument, provided the time of reading is recorded with the hardness figure. It is to be understood that, when no time is specified, the immediate reading shall be used in the indentation determination.

# *Tentative Methods of Test for*

## COMPRESSION SET OF VULCANIZED RUBBER<sup>1</sup>



A.S.T.M. Designation: D 395 - 46 T

ISSUED, 1935; REVISED, 1936, 1937, 1940, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods cover the procedures for testing vulcanized rubber parts which are to be assembled in such a manner that the rubber will be subjected to compressive stresses or shear. They are applicable particularly to the rubber used in machinery mountings and vibration dampers.

### Compression Set

2. For the purpose of these tests, compression set of rubber shall be considered as the residual decrease in thickness, expressed as the percentage of the original thickness, of a test specimen measured 30 min. after removal from a suitable loading device in which the specimen had been subjected for a definite time to compressive deformation under specified conditions of load application and temperature.

### Types of Tests

3. (a) Compression set tests are intended to measure the ability of rubber compounds to retain elastic properties

during prolonged action of compressive stresses. The actual stressing in service may involve the maintenance of a definite deflection, the constant application of a known load, or the rapidly repeated deformation and recovery resulting from intermittent compressive forces. Though the latter dynamic stressing, like the others, produces compression set, its effects as a whole are more closely simulated by compression flexing or hysteresis tests. Therefore, compression set tests are considered to be only those involving static loading.

(b) These methods cover the following two test procedures using different types of load application:

*Method A* for compression set of vulcanized rubber under constant load.

*Method B* for compression set of vulcanized rubber under constant deflection.

(c) The choice of method is optional but consideration should be given to the nature of the service for which correlation of test results may be sought. Notation shall be made in each test of the method actually used. In case of conflict between the provisions of these methods and those of detailed

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

specifications or methods of test for a particular material, method B shall take precedence.

### Apparatus

4. (a) *Compression Devices*.—The compression devices as specified in Section 7 (a) or (b) for method A and in Section 10 for method B shall be used.

(b) *Plates*.—The plates between which the test specimen is compressed shall be made of steel of sufficient thickness to withstand the compressive stresses without bending. The surfaces against which the specimen is held shall have a highly polished chromium-plated finish and shall be thoroughly cleaned and wiped dry before each test.

(c) *Oven*.—Any well designed, uniformly heated standard dry-air oven of sufficient size to hold the compression device may be used for this test. The oven shall be provided with proper temperature control to maintain the specified temperature within a permissible variation of plus or minus 1 C. (1.8 F.). Satisfactory circulation of the air shall be secured by means of a fan. The compression device shall be centrally located in the oven and supported above the bottom of the oven by means of a wire rack mounted on wood or asbestos block so that it is heated by indirect heat.

(d) *Dial Gage*.—A dial gage graduated in thousandths of an inch having a raised platform  $\frac{3}{8}$  in. in diameter and a spherical presser foot  $\frac{1}{4}$  in. in diameter, actuated by a load of  $3 \pm 0.1$  oz., shall be provided for measuring the thickness of the specimen before and after loading.

### Standard Test Specimens

5. The standard test specimen shall be a cylindrical disk  $\frac{1}{2}$  in. in thickness, cut from the vulcanized rubber part by means of a sharp, circular die having an inside diameter of  $1.129 \pm 0.001$  in. In cutting the specimen, the die shall be

suitably rotated in a drill press or similar device and lubricated by means of soapy water so that a smooth-cut surface having square edges is obtained. The cutting pressure shall be kept sufficiently small to avoid "cupping" of the cut surface. When necessary, the specimen thickness shall be reduced to the required  $\frac{1}{2}$  in. by cutting transversely with a sharp knife and carefully buffing to size within a permissible variation of plus 0.005 in. and minus 0.025 in. The buffing shall be lightly done to avoid overheating and care shall be taken to keep the circular faces parallel and at right angles to the axis of the cylinder.

### Special Test Specimens

6. In routine production testing it is sometimes more convenient to cut test specimens of a different size and shape than that specified in Section 5. When any other size of specimen is used the test results shall not be compared with those secured using the standard test specimen. When using specimens of special sizes or shapes the results may be compared only with those from similar sizes and shapes.

#### *Method A. Compression Set Under Constant Load*

### Compression Devices

7. The compression device shall consist of a loading spring and two parallel compression plates assembled by means of a frame or threaded bolts in such a manner that the device shall be portable and self-contained after the load has been applied and that the parallelism of the plates shall be maintained. The load may be applied as described in either Paragraph (a) or (b).

(a) *Calibrated Spring Loading*.—In the case of calibrated spring loading, the required load shall be applied by a screw mechanism for compressing a calibrated spring the proper amount as



measured to the nearest hundredth of an inch. The spring shall be of properly heat-treated spring steel with ends ground square and perpendicular to the longitudinal axis of the spring. A suitable compression device is shown in Fig. 1. The spring shall conform to the following requirements: (1) The spring shall be calibrated at room temperature

expressed in pounds by the difference between the corresponding deflections expressed in hundredths of an inch.

Note: *Example.*—A spring which deflects 1.00 in. at 400 lb. and 0.75 in. at 300 lb. gives a slope of 4.0.

(2) The original dimensions of the spring shall not change due to fatigue by

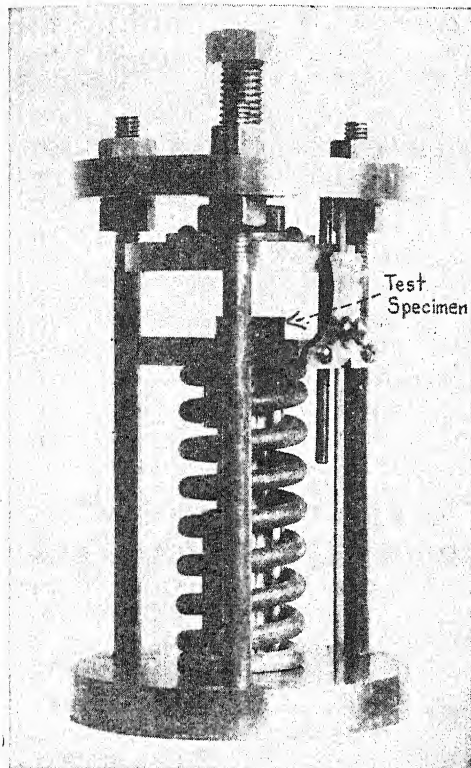


FIG. 1.—Device for Compression Set Test, Using Calibrated Spring Loading, Method A.

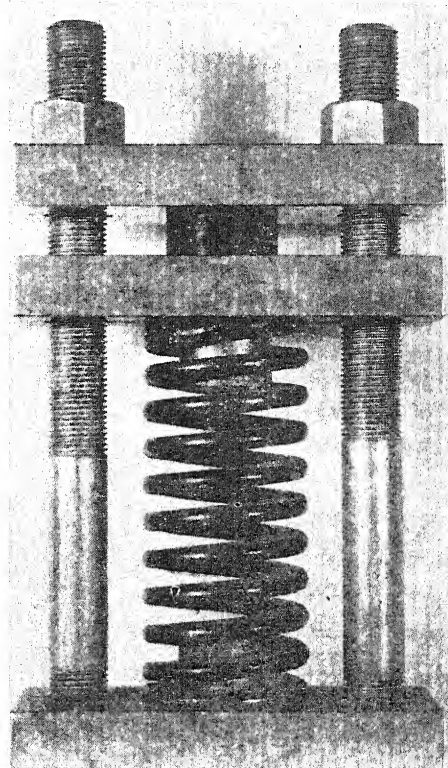


FIG. 2.—Device for Compression Set Test, Using External Loading, Method A.

( $24 \pm 5$  C. ( $75 \pm 9$  P.)) by applying successive increments of load of  $50 \pm 0.5$  lb. and measuring the corresponding deflection in hundredths of an inch. The curve obtained by plotting the loads against the corresponding deflections shall have a slope of  $4.0 \pm 0.2$  at 400-lb. load. The slope is obtained by dividing the difference between two loads

more than 0.01 in. after it has been mounted in the compression device, compressed under a 400-lb. load and heated in the oven for 1 week at 70 C. (158 F.). In ordinary use, a weekly check of the dimensions shall show no greater change than this over a period of 1 yr.

(3) The minimum load required to close the spring solid shall be 530 lb.

(b) *External Loading.*—In the case of external loading, the required load shall be applied to the compression plates and spring by external means after the test specimen is mounted in the apparatus. Either a calibrated compression machine or dead weights may be used for load application. Provision shall be made by the use of bolts and nuts or other devices to prevent the specimen and spring from losing their initial deflections when the external load is removed. The spring shall have essentially the same characteristics as described in Paragraph (a), but calibration is not required. A suitable compression device is shown in Fig. 2.

#### Procedure

8. (a) *Original Thickness Measurement.*—The original average thickness of the test specimen shall be measured to the nearest 0.001 in. The specimen shall be placed on the small platform of the dial gage so that the presser foot will indicate the thickness at the central portion of the top and bottom faces.

(b) *Application of Load.*—The test specimen shall be assembled in the compression device, using extreme care to place it exactly in the center between the plates so that tilting may be avoided. If the calibrated spring device (Fig. 1) is used, the load shall then be applied by tightening the screw until the deflection as read from the scale is equivalent to that shown on the calibration curve for the spring as corresponding to a load of 400 lb. With the external loading device (Fig. 2), a 400-lb. load shall be applied to the assembly in the compression machine or by dead weight, but in the latter case care shall be taken to apply the weight gradually without shock. The nuts and bolts shall then be tightened just sufficiently to hold the initial deflections of the specimen and spring. It is imperative that no addi-

tional load be applied in tightening the bolts.

(c) *Heat Treatment.*—Before inserting the test specimen in the compression device, the compression device shall be preheated for a sufficient length of time to insure that the entire assembly is at the testing temperature. A minimum amount of time shall be consumed in changing or in inserting test specimens. Should it be necessary to keep the device out of the oven longer than 20 min., it is suggested that the device after loading shall be immersed in water at 158 F. for a period of 20 min. before being replaced in the air oven. The assembled compression device shall be placed in the oven within 2 hr. after completion of the assembly and shall remain there for 22 hr. in dry air at 70 C. (158 F.). At the end of the heating period the device shall be taken from the oven and the test specimen removed immediately and allowed to cool.

(d) *Cooling Period.*—While cooling, the test specimen shall rest on a wooden table top for 30 min. before the measurement of the final thickness is made.

(e) *Final Thickness Measurement.*—After the rest period, the final thickness shall be measured at the center of the test specimen using the dial gage described in Section 4 (d).

(f) *Check Tests.*—Tests shall be run in duplicate but only one test specimen shall be tested at one time in each compression device. The duplicate test results should agree within 5.0 per cent of the values obtained.

#### Report

9. The report shall include the following:

(a) The original dimensions of the test specimen, including the original thickness,  $t_0$ ,

(b) The actual compressive load on the

specimen as determined from the calibration curve of the spring and the spring deflection reading (Section 7 (a)) or as applied by external loading (Section 7 (b)),

(c) The thickness of the test specimen 30 min. after removal from the clamp,  $t_1$ , and

(d) The compression set in inches and expressed as a percentage of the original thickness, calculated as follows:

$$\text{Compression set} = t_o - t_1$$

$$\text{Percentage compression set} = \frac{t_o - t_1}{t_o} \times 100$$

#### Method B. Compression Set Under Constant Deflection

##### Compression Device

10. The compression device shall consist of two or more flat steel plates between the parallel faces of which the specimens may be compressed as shown in Fig. 3. Steel spacers for the required

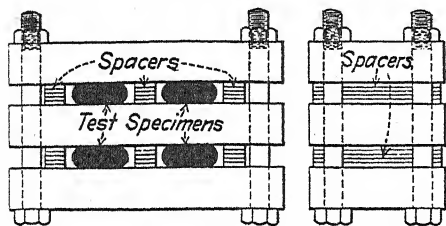


FIG. 3.—Device for Compression Set Test Under Constant Deflection, Method B.

percentage of compression given in Section 11 (b) shall be placed on each side of the rubber specimens to control their thickness while compressed. The steel surfaces contacting the rubber specimens shall be ground to a maximum roughness of 10 micro-inches and then chromium plated and polished. They shall be thoroughly cleaned before each use.

##### Procedure

11. (a) *Original Thickness Measurement.*—The original average thickness of the specimen shall be measured as described in Section 8 (a).

(b) *Application of Load.*—The test specimen shall be placed between the plates of the compression device with the spacers on each side of it. If only one specimen is being tested, it shall be placed in the center. If two specimens are being tested, three spacers shall be provided, one in the center and one outside of each specimen, allowing sufficient clearance for the bulging of the rubber when compressed (Fig. 3). The bolts shall be tightened so that the plates are in contact with the spacers. The percentage of compression employed shall depend upon the hardness of the rubber being tested, according to the following table:

Durometer Number	Hardness	Deflection, per cent of original thickness
30 ± 5	.....	40
40 ± 5	.....	40
50 ± 5	.....	30
60 ± 5	.....	30
70 ± 5	.....	25
80 ± 5	.....	25
90 ± 5	.....	20

(c) *Heat Treatment.*—The heat treatment of the specimen shall be performed in accordance with Section 8 (c).

(d) *Cooling Period.*—The specimen shall be cooled in accordance with Section 8 (d).

(e) *Final Thickness Measurement.*—The final thickness of the specimen shall be measured as described in Section 8 (e).

(f) *Check Tests.*—Tests shall be run in duplicate and the results should agree within 5.0 per cent of the values obtained.

##### Report

12. The report shall include the following:

(a) The original dimensions of the test specimen including the original thickness,  $t_o$ ,

(b) The percentage compression of the specimen actually employed,

(c) The thickness of the test specimen 30 min. after removal from the clamp,  $t_1$ , and

(d) The compression set expressed as a percentage of the original deflection shall be calculated as follows:<sup>3</sup>

$$C = \frac{t_0 - t_1}{t_0 - t_s} \times 100$$

where:

$C$  = compression set expressed as a percentage of the original deflection, and

$t_s$  = thickness of the spacer bar used.

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<sup>3</sup> It has been found that the compression set as calculated in Section 12 (d) is substantially constant for specimens cut from any one sample regardless of the original deflection (within the limits of the table in Section 11 (b)) actually used.

# *Tentative Methods of Test for*

## COMPRESSION FATIGUE OF VULCANIZED RUBBER<sup>1</sup>



A.S.T.M. Designation: D 623 - 41 T

ISSUED, 1941.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods are intended for use in comparing the rate of heat generation and the fatigue characteristics of vulcanized rubber compounds that may be subjected in use to dynamic compression strains. Owing to wide variations often present in service conditions, no direct correlation between these accelerated tests and service performance may be given or implied. However, the methods yield comparative data on which to base judgment as to service quality, and are especially useful in research and development work. They are not suitable for use in purchase specifications, because both correlation with service life and reproducibility between different laboratories are uncertain. It is assumed that the methods will be used principally for intercomparisons between compounds tested under identical conditions, in which case the compounds showing lowest heat generation and least change due to fatigue may be considered superior for those service applications where these factors are considered significant.

### Types of Test

2. (a) In general, the test consists of subjecting a specimen of rubber of definite size and shape to rapidly oscillating compressive stresses under controlled conditions and measuring the extent and rate of the temperature rise of the specimen, the impressed loads, the dimensional changes, and the time required for failure by actual blowout, if this occurs.

(b) These methods cover three test procedures using the following different types of apparatus:

Method A. The Goodrich Flexometer.

Method B. The Firestone Flexometer.

Method C. The St. Joe Flexometer.

### Preparation of Sample

3. (a) The sample may consist of any vulcanized rubber compound except those generally classed as hard rubber, provided it is of sufficient size to permit preparation of the test specimen required for the method to be employed. The sample may be prepared from stock mixed experimentally in the laboratory or taken from process during manufacture, or it may be cut from a finished article of commerce.

(b) If prepared in the laboratory, the

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1941.

procedure shall be essentially as specified in Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society for Testing Materials,<sup>3</sup> except that when vulcanization is required, the sample shall be molded in block form of sufficient size to permit cutting of the required test specimens rather than in the form of the standard test slab.

(c) Samples from commercial articles shall consist of a piece slightly larger than the required test specimen and shall subsequently be cut or buffed to size.

(d) Comparison of results shall be made only between specimens of identical size and shape.

#### METHOD A. GOODRICH FLEXOMETER<sup>4</sup>

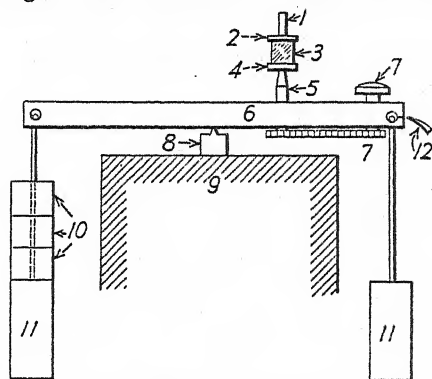
##### Nature of Test

4. In this method, which uses the Goodrich Flexometer, a definite compressive load is applied to a test specimen through a lever system having high inertia, while imposing on the specimen an additional high-frequency cyclic compression of definite amplitude. The increase in temperature at the base of the test specimen is measured with a thermocouple to provide a relative indication of the heat generated in flexing the specimen. Specimens may be tested under a constant applied load, a constant initial compression, or a constant deflection during the test. The change in height of the test specimen can be measured continuously during flexure. By comparing this change in height with the observed permanent set after test, the degree of stiffening (or softening) of the test specimen may be estimated. *Anisotropic* specimens may be

tested in different directions producing measurable differences in temperature rise due to the *anisotropy*.

##### Apparatus

5. (a) The essential parts of the apparatus are shown in Fig. 1. The test specimen is placed between anvils covered with special heat-resistant hard rubber insulators. The top anvil is connected to an adjustable eccentric driven at 1800 rpm. The load is applied by means of a lever resting on a knife-edge. The moment of inertia of the



- |   |   |
|---|---|
| 1—Connection to eccentric which drives top anvil. | 7—Calibrated micrometer device.                                 |
| 2—Top anvil.                                      | 8—Knife-edge.   |
| 3—Test specimen.                                  | 9—Supporting base.  |
| 4—Lower anvil.                                    | 10—Test load.   |
| 5—Support for lower anvil.                        | 11—Equal 50-lb. weights for increasing inertia of lever system. |
| 6—Lever through which load is applied.            | 12—Pointer and reference mark for leveling of lever.            |

FIG. 1.—Goodrich Flexometer.

lever system is increased, and its natural frequency reduced, by suspending equal weights of 50 lb. at each end of the lever at positions equidistant from the knife-edge. The lower anvil can be raised and lowered relative to the lever by means of a calibrated micrometer device. With this device the bar may be maintained in a horizontal position during a test, as indicated by a pointer and a reference mark on the bar. The increase in temperature at the base of the specimen is determined by means of a thermo-

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> E. T. Lessig, *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 9, pp. 582-588 (1937).

couple placed at the center of the bottom anvil. To keep the specimen in contact with the insulated anvils during the entire compression cycle, the total length of the compression stroke shall be less than about twice the initial compression of the test specimen. The length of the compression stroke is adjusted by means of a vernier and scale on the eccentric.

(b) The machine may be equipped with a well-insulated oven surrounding the test specimen so as to permit running of tests at elevated temperatures. The oven is controlled by means of a thermostat and is provided with a small motor-driven fan to maintain air circulation. Use of the oven also assures uniform temperature of the air surrounding the specimen.

### Test Specimen

6. The test specimen shall be cylindrical in shape, 1 in. in height and 0.7 in. in diameter, and may be conveniently cut from larger blocks or from manufactured articles, using a cylindrical cutting tool either in a drill press or lathe. Laminated test pieces also may be built up without the use of adhesive from thin disks 0.7 in. in diameter arranged horizontally in the machine.

### Procedure

7. (a) Before starting a test, the eccentric which drives the top anvil shall be adjusted to give the desired length of stroke. The top anvil shall then be raised as far as the eccentric will permit and the micrometer device adjusted by inserting a calibrating block 1 in. in height between the anvils and setting the micrometer at zero. If necessary, adjustment of the cross bar holding the top anvil shall be made to bring the lever bar to a horizontal position. The pointer shall then be set on the mark on the end of the lever bar to mark the zero position.

(b) After removing the calibrating block, the test specimen shall be placed between the anvils, properly centered over the thermocouple, and loaded by releasing the lever bar on which the desired loading weight has been suspended. The bar shall then be returned to its zero position by turning the micrometer mechanism and the static compression of the specimen under load shall be read directly on the micrometer scale which is graduated to 0.001 in. The lever bar shall then be fastened in the zero position and the motor which drives the eccentric started, after which the bar shall be again allowed to act freely on the specimen and shall be readjusted to its zero position while the specimen is being flexed. This readjustment shall be repeated at intervals during the test to indicate on the micrometer the change in the mean height of the specimen during flexure.

(c) The increase in temperature, measured at the base of the specimen by means of the thermocouple, shall be observed progressively during the test. This depends not only on the heat generated but also on the conductivity and emissivity of the test specimen, the conductivity of the insulators,<sup>5</sup> and other factors. Corrections for differences in conductivity may be made, but in practice may be neglected and stocks compared on a basis of actual temperature rise at the base of the test specimen.

(d) The duration of the test is dependent on the information desired as well as on the type of stock being tested. Tests may be made for a definite time interval, until there is no further increase in heat build-up, or until actual

<sup>5</sup> Thermal and insulating materials other than those specified and supplied with the machine may be substituted, provided that their thermal conductivity, specific heat, specific gravity, and softening temperatures be the same within plus or minus 2½ per cent of the respective values for the specified materials. Detailed drawings for the anvil construction are on file at Society Headquarters.



blowout of the specimen occurs. In practice, the length of stroke usually chosen may vary from 0.175 to 0.375 in. and the added load on the lever bar may be such as to give from 100 to 300 psi. on the specimen. The longer strokes and heavier loads shorten the duration of the test before maximum temperature rise or actual blowout occurs.

- (1) Description of test specimen,
- (2) Applied static load and length of stroke,
- (3) Initial temperature of specimen,
- (4) Initial compression as a percentage of the original height,
- (5) Observed maximum temperature rise in degrees Fahrenheit,
- (6) Duration of test,

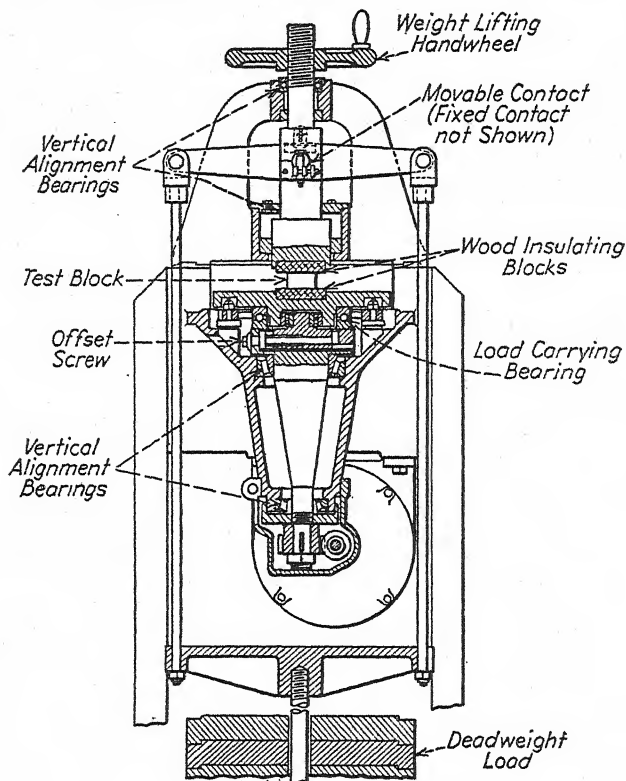


FIG. 2.—Firestone Flexometer.

(e) The permanent set shall be taken as the percentage difference in height between the original unstressed test specimen at room temperature and the height of the test specimen rested at least 1 hr. at room temperature after flexure, referred to the original height.

#### Report

8. The report shall include the following:

- (7) Notation whether blowout occurred, and
- (8) Permanent set as a percentage of original height.

#### METHOD B. FIRESTONE FLEXOMETER<sup>6</sup>

##### Nature of Test

9. In this method, which uses the Firestone Flexometer, a rotary motion

<sup>6</sup> L. V. Cooper, *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 5, pp. 350-351 (1933).

is applied to one end of a test specimen held under a constant compression load, and the time required for a definite change in height of the test specimen is determined.

### Apparatus

10. The apparatus is illustrated in Fig. 2. The speed of oscillation of the oscillating plate shall be constant at 800 cycles per min., but the compression load and the magnitude of the oscillation may be varied over a wide range. The oscillating and loading plates are equipped with center inserts of wood, 3 in. (7.62 cm.) in diameter and 0.50 in. (1.27 cm.) in thickness.

### Test Specimen

11. The laboratory test specimen shall be in the shape of a frustum of a rectangular pyramid and shall have the following dimensions: base, 2.125 by 1.125 in. (5.40 by 2.86 cm.); top, 2 by 1 in. (5.08 by 2.54 cm.); and altitude, 1.50 in. (3.81 cm.). This tapered shape permits the preparation of perfect specimens from any type of stock. Test specimens of cured articles may be cut into any suitable size and shape, provided a similar specimen of a known control may be prepared.

### Procedure

12. (a) The oscillating plate shall be set on dead center and brought to a definite starting temperature.

(b) The test specimen shall be placed on the oscillating plate, directly under the loading plate and between the wood inserts in the plates. The wood inserts act as heat insulators, and tend to hold all generated heat in the test specimen.

(c) The load shall be applied to the test specimen by turning the hand wheel on top until the load is carried by the

block and not by the thrust bearing on the wheel.

(d) The height of the block after applying the load shall then be measured, and the deflection calculated.

(e) The oscillating plate shall be set the desired amount off center, distorting the test specimen so that it resembles the frustum of a sloping rectangular pyramid. The diameter of the circle described by the lower plate while in motion is designated as the "throw."

(f) The electrical bell-ringing contacts shall be adjusted so that they are a definite distance apart.

NOTE.—This initial contact opening is called "signal distance." As the block is deflected under the testing conditions, the upper contact is carried downward toward the lower contact, because the load, which is a dead weight, continues to rest on the yielding block. This downward movement has been found by many tests to be a definite criterion of the condition of the center of the block. In other words the signal distance, at the time porosity began, was the same for all blocks of similar composition. Should the test be continued sufficiently long, the block will actually blow out, or shatter to pieces, and it is to prevent this actual destruction that the yield distance of slight porosity is used.

(g) The duration of the test, which is now ready to be started, shall be measured from the time the circular motion is started until the bell rings. The bell does not ring until the test block has been deformed sufficiently to allow the electrical contact to close.

### Precautions

13. Care should be taken that only well-molded blocks are used and that the machine is correctly set each time. If these two precautions are taken, consistent results will be obtained. The machine is not intricate in design or movement, yet it can readily be used for a wide variety of tests.

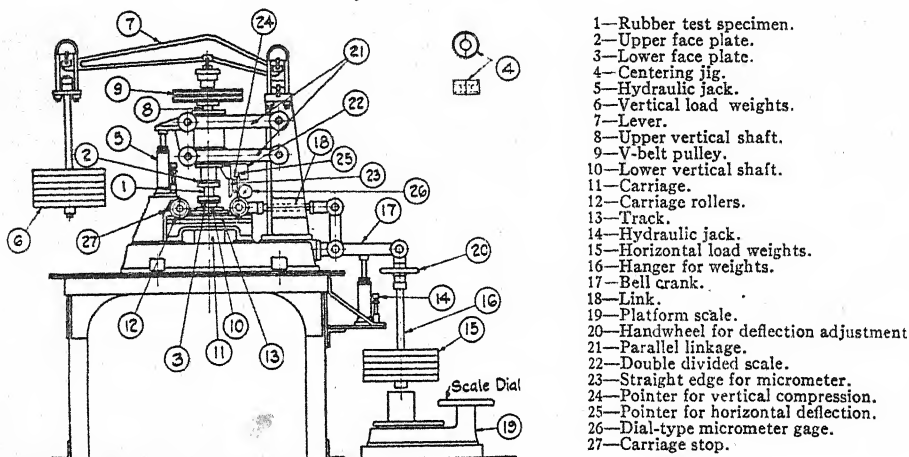
METHOD C. THE ST. JOE FLEXOMETER<sup>7</sup>

## Apparatus

## Nature of Test

14. In this method, which uses the St. Joe Flexometer, the rate of heat generation and breakdown characteristics of rubber compounds when flexed under compressive loads are measured. The machine is designed to determine the point at which incipient failure starts

15. The essential features of the apparatus, illustrated in Fig. 3, are as follows: The test specimen of rubber acts as a coupling between the two parallel face plates, which are mounted on concentric vertical shafts in such a manner that the rotation of the top plate is transmitted through the specimen to the



Description of Machine.

Figure 3, a drawing of the complete machine, shows the mechanism for loading and flexing the test specimen, as well as the means by which the flexure and flexing forces are measured.

Test specimen 1 is accurately positioned between face plates 2 and 3 through use of centering jig 4. By release of jack 5, the load is applied vertically to the test specimen from

weights 6, acting through lever arm 7, which rests on top of vertical shaft 8, producing compression in the test specimen. This shaft is driven by pulley 9 which is belted to a vertical shaft motor. The lower face plate 3 rests on vertical shaft 10 and is driven by the upper shaft and face plate through the test specimen, which acts as a coupling. Vertical shaft 10 and carriage 11 are mounted on rollers 12 that ride on track 13.

FIG. 3.—St. Joe Flexometer.

in the center of the test specimen. However, the test can be continued and the time and loadings for actual heat blow-out can also be recorded.

lower plate. The upper plate shall be motor driven at 875 rpm.<sup>8</sup> A vertical load shall be applied to the test specimen through a lever arm acting on the top face plate so that the specimen is compressed while rotating between the two plates. At the same time a horizontal

<sup>7</sup> This method was originated by the St. Joseph Lead Co. For further information respecting this test, see R. S. Havenhill and W. B. MacBride, "A New Laboratory Machine for Evaluating Breakdown Characteristics of Rubber Compounds," *Industrial and Engineering Chemistry*, Vol. 7, No. 1, January 15, 1935, p. 60; also R. S. Havenhill, "Heat Generation and Anisotropy of Rubber Compounds in Flexometer," *Physics*, Vol. 7, No. 5, May, 1936, p. 197; also "Evaluating Carbon Black for the Rubber Industry in the Huber Laboratory," p. 24, J. M. Huber, Inc., (1936.)

<sup>8</sup> A constant operating speed is essential; where line voltages are variable, suitable precautions must be taken to obtain constant operating speed.

load shall be applied to the lower face plate, thus moving the bottom of the specimen off center. The eccentricity of the two rotating plates produces a flexing of the rotating specimen while it is under compression, and results in a sharply defined blowout or failure. The end point of the test is the time and loading required to produce incipient failure, which is indicated on the horizontal load scale by an abrupt dropping off in horizontal load, that is, flexing load. The vertical load, vertical compression, horizontal flexing load, and horizontal deflection may all be measured and controlled during test. Temperature measurements on specimens at various period of test may be obtained by means of a modified hypo needle thermocouple.

**NOTE.**—It is readily apparent that either constant load or constant deflection type tests can be made and that time for failure can be varied from 1 min. to several months by varying either the vertical load or horizontal deflection; however, it is desirable to have a test period of 30 to 60 min., such time being long enough to obtain good differentiation between specimens and short enough not to entail excessive cost. It is also desirable to operate the machine at constant vertical load and constant horizontal deflection, and to measure the resulting changes in vertical compression and horizontal flexing loads, as well as the time for incipient failure.

### Test Specimens

16. (a) The test specimens when prepared in the laboratory shall be cylindrical in shape and shall be 1.5 in. in diameter by 1.5 in. in height. Smaller cylindrical specimens may be used when samples are cut from finished tire treads or motor mountings.

(b) Test specimens shall be prepared from uncured stock which shall be sheeted out on a 6 by 12-in. laboratory mill to a thickness of 0.12 in. Strips 2.0 in. in width shall be cut off and rolled into cylinders approximately 1.45

in. in diameter with the grain of the rubber parallel to the axis of the cylinder. Grain direction shall be maintained in the same direction to assure duplication of results. Care shall be taken to insure that no dust, crayon markings, or other foreign material, which would prevent proper cohesion of layers, are on a sheet before it is rolled up.

(c) The cylindrical specimens shall be cut to weight, aged 3 hr., then cured. The cured stocks shall be aged 24 hr. prior to testing. At least two specimens from each rubber stock shall be tested and the results averaged. It is desirable to mix, cure, and test simultaneously with each set of specimens a control stock. For convenience, an eight-cavity mold may be used.

### Procedure

17. (a) The test specimen shall be accurately centered between the upper and lower face plates of the properly aligned machine, by means of the jig furnished with the machine. The temperature of the plates<sup>9</sup> shall be 128 F. and the machine shall be at equilibrium temperature at the start of each test.

(b) The vertical load shall be applied and the test specimen rotated at 875 rpm. The vertical load shall be so chosen that it will produce in the specimen a vertical deflection of 0.35 to 0.80 in. It is desirable to have a vertical load that will duplicate service conditions as near as is possible keeping within this specified vertical deflection. A vertical load of 575 lb. for tread stocks and of 400 lb. for carcass stocks is suitable. The same vertical load shall be used for all specimens when a comparison of their flexing performance is made. The hori-

<sup>9</sup> The temperature of the face plates has a marked effect on the results, so that it is necessary, prior to any testing, to run a dummy specimen to bring them to operating temperature. Furthermore, the machine shall be run for 1 hr. before any tests are made, so that the upper head containing the rotating spindle and the lower carriage will come to equilibrium temperature.

zontal load shall then be applied until the horizontal deflection is such a value as to produce failure in 30 to 60 min. The deflection necessary to produce such failure shall be determined by preliminary tests. A horizontal deflection<sup>10</sup> of  $0.175 \pm 0.001$  in. for tread stocks is usually suitable. The same horizontal deflection shall be used for all specimens when a comparison of their flexing performance is made. This deflection shall be maintained constant throughout the test by means of the handwheel. Readings of vertical compression, horizontal deflection, and horizontal or flexing load shall be recorded at intervals of 2 min. until failure occurs, which shall be taken as the time at which an abrupt drop in horizontal load is observed.

### Results

18. The time and flexing load required to cause breakdown of the test specimen

<sup>10</sup> This deflection will vary for different type stocks but shall be chosen so that a 30 to 60-min. breakdown of the stock shall be obtained when possible.

shall be taken as the time in minutes and flexing load in pounds required to produce incipient failure, as indicated by an abrupt drop in horizontal (flexing) load, the horizontal deflection remaining constant.

NOTE—For special tests, the time and flexing load required to produce either definite vertical compression or definite change in vertical compression may be recorded. The time and flexing load for blowout or complete rupture may also be recorded.

### Report

19. The report of the results of tests by either Methods B or C shall include the following:

(1) The value of all variables in the method such as speed of testing, vertical and horizontal loads, and air or oven temperatures.

(2) All measured results such as changes in temperature of the test specimen, changes in static or dynamic compression, and time to end point, and all other pertinent observations.

## *Tentative Method of Test for*

### RESISTANCE OF VULCANIZED RUBBER OR SYNTHETIC ELASTOMERS TO CRACK GROWTH<sup>1</sup>



A.S.T.M. Designation: D 813 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. This method of test is intended for use in determining the resistance of compounds of vulcanized rubber or synthetic elastomers to crack growth when subjected to repeated bend flexing. It is particularly applicable to tests of synthetic rubber compounds which resist the initiation of cracking due to flexing when tested by Method B of the Standard Methods of Dynamic Testing for Ply Separation and Cracking of Rubber Products (A.S.T.M. Designation: D 430).<sup>3</sup> Cracking initiated in these materials by small cuts or tears in service may rapidly increase in size and progress to complete failure even though the material is extremely resistant to the original flexing-fatigue cracking. Because of this characteristic of synthetic compounds, particularly those of the GR-S type, this method in which the specimens are first artificially punctured in the flex area should be used

in evaluating the fatigue cracking properties of this class of material.

#### De Mattia Flexing Machine

2. The essential features of the apparatus, one design of which is shown in Fig. 1, are as follows: The machine has an adjustable stationary head or member provided with suitable grips for holding one end of each of the test specimens in a fixed position and a similar reciprocating member for holding the other end of each of the specimens. The reciprocating member is so mounted that its motion is straight in the direction of and in the same plane as the center line between the grips. The travel of the moving member shall be adjustable and shall be obtained by means of a connecting rod and eccentric having a minimum length ratio of 10 to 1. The eccentric shall be driven by a motor operating at constant speed under load and giving 300 flexing cycles per minute. Provision shall be made for a maximum travel of the moving grips of 4 in. The capacity of the machine shall be such that tests at the same time may be made on at least six and preferably

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, December 13, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

twelve specimens. The grips shall hold the specimens firmly throughout the test and those on the reciprocating member may clamp each specimen individually to facilitate proper adjustment of the specimens.

### Test Specimens

3. The test specimens shall be special molded strips, conforming to the shape and dimensions given in Fig. 2. They shall have highly polished surfaces and shall be free from surface irregularities

### Preparation of Specimen

5. Each test specimen shall be prepared by puncturing the bottom of the groove at the 90 deg. point with a needle 0.05 in. in diameter at a point equidistant from the sides. The needle shall be drawn completely through the specimen. The puncturing shall be done with the specimen lying flat, under no tension.

### Clamping Specimens in Machine

6. One end of the specimen shall be clamped in the stationary grip and the

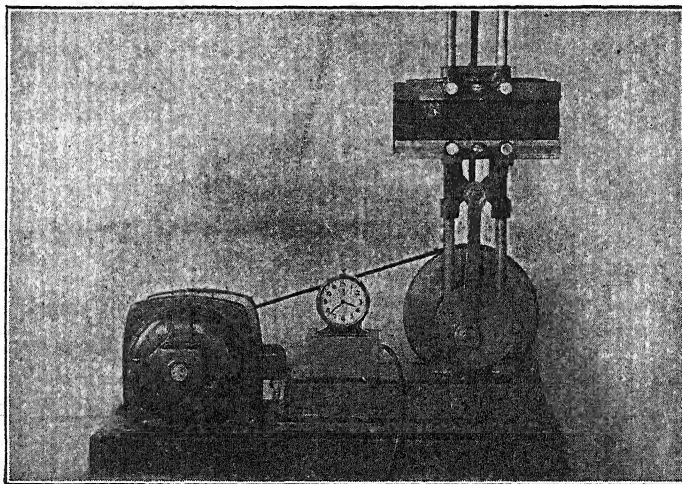


FIG. 1.—De Mattia Tester with Time-Switch for Starting or Stopping, Arranged with Specimens for Flex-Cracking Test.

and defects. The minimum thickness in the circular groove shall be measured to the nearest thousandth of an inch and recorded. Results shall be compared only between specimens having thicknesses agreeing within plus or minus 0.0015 in.

### Number of Test Specimens

4. At least three specimens of each sample shall be tested and the results averaged. It is desirable, when possible, to test simultaneously with each set of specimens a set of control specimens of which the crack growth resistance is known.

other in the movable grip, care being taken to see that the long axis of the specimen is parallel to the direction of motion. The circular groove of the clamped specimens shall be located symmetrically midway between the clamps. The specimens may be mounted on the machine most conveniently by holding them properly spaced in parallel positions in a special rack. The distance between the outer edges of the side bars of the rack shall be equal to the space between the jaws of the testing machine when positioned for holding the specimens without tension. The specimens can be mounted on the testing machine by bringing the



jaws into contact with the mounting rack and tightening the clamps on the projecting ends of the specimens. The clamps shall grip each end over a length of  $1\frac{3}{8}$  in. with the side having the circular groove so placed that it will be the outer surface when the specimens are bent.

#### Adjustment of Machine

7. The positions of the stationary and movable grips relative to each other and the length of the eccentric arm and con-

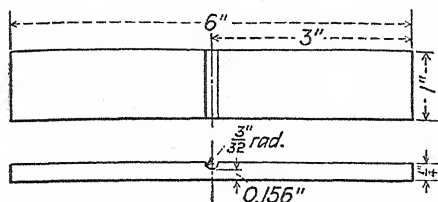


FIG. 2.—Test Specimen with Circular Groove.

necting rod shall be adjusted so that during each stroke of the machine the grips approach each other to a distance of  $\frac{3}{4}$  in. and separate to a distance of 3 in.

#### Procedure

8. After adjustments of the apparatus and specimens have been completed, the machine shall be started and the time recorded. At the end of any period of

operation, the number of flexing cycles may be calculated by multiplying the observed time in minutes by the machine rate of 300 cycles per minute. This should also be checked by means of a counter on the machine. Since the rate of crack growth is important, frequent readings should be taken early in the test. The machine shall be stopped at the 1000, 3000, and 5000 cycle periods, the specimens observed and the length of the developed crack measured to the nearest 0.01 in. with an accurate scale. The test shall then be continued with readings at regular intervals until a crack at least 0.50 in. in length is developed. When testing aged specimens or in special tests at elevated temperature, it may be necessary to continue the test until the crack is 1.0 in. in length in order to obtain an accurate evaluation.

#### Report

9. The crack growth at the conclusion of the test may be reported in one of two ways, as follows:

(1) As the individual rate of crack growth in inches per kilocycle determined between the intermittent readings taken during the test, or

(2) As an average rate of crack growth in inches per kilocycle for the entire period.

# *Tentative Methods of Test for* CHANGES IN PROPERTIES OF RUBBER AND RUBBER-LIKE MATERIALS IN LIQUIDS<sup>1</sup>



A.S.T.M. Designation: D 471 - 46 T

ISSUED, 1937; REVISED, 1940, 1943, 1944, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods are intended for use in estimating the comparative ability of rubber and rubber-like compositions to withstand the effect of liquids. Owing to the wide variations often present in service conditions, no direct correlation between these accelerated tests and service performance may be given or implied. However, the methods yield comparative data on which to base judgment as to service quality and are especially useful in research and development work. They are designed for testing any vulcanized rubber or rubber-like composition in the form of standard laboratory test sheets and should not be used for samples cut from a finished article of commerce. It is assumed that the tests will be used principally for intercomparisons between compounds rather than for an estimation of service performance of manufactured products.

## Types of Test

2. (a) This test consists in exposing test specimens to the influence of liquids under definite conditions of temperature and time for the purpose of measuring the resulting deterioration. The type of test varies depending upon the conditions of test and the nature of the liquid used. In any case, the deterioration is measured by the change in physical properties before and after immersion in the liquid. The physical properties used are changes in weight, volume, tensile strength, and ultimate elongation, as well as visual and manual inspection.

(b) In addition to the general procedure for determining these changes, which are given in method A, there is included in method B an alternative procedure for determining increase in volume by linear measurement which, though slightly less precise than the corresponding procedure in method A, is especially suitable for rapid comparative tests of swelling when a large number of solvents or rubber compounds are involved.

<sup>1</sup> Under the standardization procedure of the Society these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.  
<sup>2</sup> Latest revision accepted by the Administrative Committee on Standards, January 10, 1946.

## Test Conditions

3. (a) *Temperature*.—A suitable test temperature shall be chosen depending upon the conditions of the expected service. It shall be equal to or higher than the anticipated service temperature. In comparative tests, identical temperatures shall be employed. Use of one of the following temperatures is recommended:  $25 \pm 5$  C.,  $70 \pm 1$  C.,  $100 \pm 1$  C.

(b) *Periods of Immersion*.—Choice of the period of immersion to be used in any given test depends upon the nature of the composition, the temperature of test, and the liquid employed. Periods of 24 hr., 48 hr., 7 days, and 30 days are suggested as suitable, but it is recommended that determinations be made and recorded after several periods of immersion in order to obtain information concerning the rate of deterioration.

(c) All immersion tests shall be made in the absence of light.

(d) Each specimen shall be placed in a separate container during the immersion test.

## Standard Test Liquids

4. For purpose of tests it is usually desirable to use the liquid with which the material to be tested will come into contact in service. For comparative tests with liquids of unknown or doubtful composition, samples of liquid from the same drum or shipment shall be used. Many commercial liquids, particularly petroleum products, are subject to sufficient variation that it is not practical to use them for test media. It is then advisable to substitute standard test media covering the range of properties that may be encountered in the particular liquid. The following test liquids shall be considered as standard:

(a) *Petroleum-Base Oils*.—When petroleum-base lubricating oils are used as test liquids, the test shall be conducted

in the medium prescribed in Table I which has its aniline point nearest that of the oil with which the rubber or synthetic rubber is expected to come in contact in service, except as indicated in Paragraph (c). The aniline point of a petroleum oil appears to characterize the swelling action of that oil on synthetic rubber. In general, as the aniline point becomes lower, the swelling action

TABLE I.—PETROLEUM-BASE OILS.<sup>a</sup>

	Petroleum-Base Oil			A.S.T.M. Methods <sup>a</sup>
	No. 1 Low Swelling, Aniline Point 124 C.	No. 2 Medium Swelling, Aniline Point 93 C.	No. 3 High Swelling, Aniline Point 70 C.	
Saybolt Universal viscosity, sec. . .	$98 \pm 5^b$	$100 \pm 5^b$	$155 \pm 5^c$	D 88
Aniline point. . .	$123.9 \pm 1$ C. ( $255 \pm 1.8$ F.)	$93 \pm 3$ C. ( $199.4 \pm 5.4$ F.)	$69.5 \pm 1$ C. ( $157.1 \pm 1.8$ F.)	D 611
Flash point. . .	470 F. min.	$475 \pm 10$ F.	$330 \pm 5$ F.	D 92

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88),<sup>4</sup> Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611),<sup>4</sup> and Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>5</sup>

<sup>b</sup> Measured at 210 F.

<sup>c</sup> Measured at 100 F.

of the oil becomes more severe. The oils prescribed in Table I cover a range of aniline points commonly found in lubricating oils.

(b) *Reference Fuels*.—It is recommended that when gasolines are used as test liquids, the tests shall be conducted in one of the Reference Fuels prescribed in Table II, except as indicated in

<sup>5</sup> The following suppliers state that their products meet the requirements for these petroleum-base oils when ordered as A.S.T.M. oils as follows; (oils No. 1 and No. 3 are available only in 5- and 55-gal. quantities):

A.S.T.M. Oil No. 1 may be ordered as such from Stanco Distributors, Inc., Chemical Products Dept., Attention Mr. A. B. Boehm, 26 Broadway, New York 4, N. Y.

A.S.T.M. Oil No. 2 may be ordered as Sun XX Heavy Cable from Sun Oil Co., Philadelphia, Pa.

A.S.T.M. Oil No. 3 may be ordered as such from Sun Oil Co., Attention Mr. J. B. Hill, Marcus Hook, Pa.

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

Paragraph (c). These test fuels have been selected to include the maximum and minimum swelling effects which will be produced by commercial gasolines. Reference Fuel No. 1 has a mild action on rubber or synthetic rubber and produces results of the same order as low-

TABLE II.—REFERENCE FUELS.<sup>6</sup>*Reference Fuel No. 1:*

Diisobutylene<sup>a</sup> containing 0.2 lb. of aviation gasoline inhibitor<sup>c</sup> per 1000 gal. of diisobutylene.<sup>f</sup>

*Reference Fuel No. 2:*

Diisobutylene <sup>a</sup> .....	60 per cent by volume
Benzene <sup>b</sup> .....	5 per cent by volume
Toluene <sup>c</sup> .....	20 per cent by volume
Xylene <sup>d</sup> .....	15 per cent by volume
Inhibitor.....	0.2 lb. of aviation gasoline inhibitor <sup>c</sup> per 1000 gal. of fuel.

<sup>a</sup> Diisobutylene conforming to the following requirements:

Test	Limits	Methods <sup>g</sup>
Gravity at 60 F....	0.718 to 0.723	A.S.T.M. D 268
Acidity.....	Neutral to phenolphthalein	
Color.....	+25 Saybolt, min.	A.S.T.M. D 156
Sulfur.....	0.06 per cent by weight	A.S.T.M. D 90
Gum.....	5 mg. per 100 ml.	Federal 330.1
Distillation.....	Initial 100 C. min. Dry point 105 C. max.	A.S.T.M. D 268

<sup>b</sup> Benzene conforming to U. S. Army Specifications 4-1016B, Grade A.

<sup>c</sup> Toluene conforming to Army-Navy Aeronautical Specifications for Toluene (AN-R-T-541).

<sup>d</sup> Xylene conforming to Army-Navy Aeronautical Specifications for Xylene (AN-R-X-876-2).

<sup>e</sup> The aviation gasoline inhibitor shall consist of 50 percent of *n*-Butyl-*p*-aminophenol, 30 percent of isopropyl alcohol, and 20 percent of anhydrous methanol.

<sup>f</sup> These concentrations in the fuels are expressed in terms of the active ingredient (*n*-Butyl-*p*-amino phenol) in the inhibitor solution.

<sup>g</sup> These designations refer to the following methods of test:

Standard Methods of Sampling and Testing Lacquer Solvents and Diluents (A.S.T.M. Designation: D 268),<sup>6</sup>

Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (A.S.T.M. Designation: D 156),<sup>4</sup>

Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (A.S.T.M. Designation: D 90),<sup>4</sup> and

Method of Test for Gum Content of Gasoline (Copper Dish Method, 330.11) in the Federal Specification for Lubricants and Liquid-Fuels; General Specifications (Methods for Sampling and Testing) (VV-L-791c).

swelling gasolines of the highly paraffinic, straight-run type. Reference Fuel No. 2 has a severe swelling action on rubber or synthetic rubber and exceeds the swelling action of gasolines now in service.

<sup>6</sup> Stanco Distributors, Inc., 26 Broadway, New York, N. Y., state that reference fuel SR-10 supplied by them conforms to the above requirements for reference fuel No. 1 and SR-6 to the requirements for reference fuel No. 2.

<sup>7</sup> 1946 Book of A.S.T.M. Standards, Part II.

(c) *Service Fluid*.—Some commercial products are compounded from special petroleum hydrocarbon fractions or mixtures of petroleum hydrocarbon fractions and other ingredients, resulting in materials having properties beyond the range of the present standards. Immersion tests for rubber or synthetic rubber which is to come into contact with such liquids should include immersion in the actual service fluid.

### Preparation of Sample

5. Except as may be otherwise specified, the requirements of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15) of the American Society of Testing Materials<sup>7</sup> shall be complied with and are made a part of these methods.

### Method A. General Procedures

#### Apparatus

6. The apparatus shall consist of the following:

(a) *For Volatile Liquids*.—A glass test tube having an outside diameter of 38 mm. and an over-all length of 300 mm., fitted tightly with a cork stopper and a reflux condenser. A suitable bumper such as glass beads shall be used in the liquid in the test tube.

(b) *For Nonvolatile Liquids*.—A glass test tube having an outside diameter of 38 mm. and an over-all length of 200 mm., fitted loosely with a cork stopper. A glass test tube having an outside diameter of 38 mm. and an over-all length of 300 mm. may be used.

#### Test Specimens

7. (a) The test specimens shall be cut longitudinally from vulcanized sheets prepared as described in Sections 4 to 6 of A.S.T.M. Methods D 15.

(b) For the determination of changes in weight or volume, and for visual or

<sup>7</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

manual inspection, rectangular test specimens having dimensions of 1 by 2 by  $0.075 \pm 0.005$  in. shall be used.

(c) For immersion in connection with the determination of changes in tensile strength and ultimate elongation (Section 10), the test specimens shall conform in shape to die C in Fig. 3 of the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials,<sup>7</sup> and shall have a thickness of  $0.075 \pm 0.005$  in.

(d) At least three test specimens shall be used for each determination and the results averaged.

#### Increase in Weight

8. The test specimen shall be weighed in air to the nearest 1 mg. ( $W_1$ ) and placed in a test tube as described in Section 6 (a) or (b), depending on the type of liquid to be used in the test. To this test tube shall be added 100 ml. of test liquid. If a nonvolatile liquid is used, the test tube may then be placed in a thermostatically controlled air oven for the required length of time. However, if a volatile liquid is used, the test tube shall be fitted with a reflux condenser as described in Section 6 (a) and placed in a liquid bath thermostatically controlled at the desired temperature of test. In either case a control test tube containing the test liquid or any other suitable medium and a precision thermometer shall be used along with the test tubes containing the test specimens. After the immersion test has proceeded the required length of time, the test specimen shall be removed, dipped quickly into SDA-2B alcohol, blotted lightly with filter paper, placed immediately in a tared, stoppered weighing bottle, and the weight after test determined ( $W_2$ ). After weighing, the test specimen shall be again immersed in the test liquid if data is desired on the pro-

gressive changes which occur with increasing time of immersion. It is important that each manipulation take place promptly with the least possible lapse of time. No more than 30 sec. shall be allowed for each step except the actual weighing in the tared, stoppered weighing bottle.

#### Increase in Volume

9. (a) *Water Displacement Method for Water Insoluble Liquids and Mixed Liquids.*—The initial water displacement of the specimen shall be determined by weighing it in air to the nearest milligram ( $W_1$ ) and in distilled water at room temperature ( $W_2$ ), the difference being the weight of the water displaced by the specimen. The specimen shall then be dipped quickly into SDA-2B alcohol, blotted dry with filter paper, and placed in the immersion apparatus as described in Section 6 (a) or (b). Then 100 ml. of liquid shall be added to the test tube and the immersion test carried out as described in Section 8. At the end of the required length of immersion time, the specimen shall be removed from the test tube and dipped quickly into SDA-2B alcohol at room temperature, blotted lightly with filter paper, placed in a tared weighing bottle, and weighed ( $W_3$ ). It shall then be removed from the bottle and weighed in distilled water ( $W_4$ ) in immediate consecutive procedure to determine the water displacement after test. If the immersion has been at an elevated temperature, the test specimen should be cooled to room temperature at the end of the immersion period by transferring it to a cool, clean portion of the test liquid prior to being dipped in alcohol. It is important that all air bubbles clinging to the test specimens be removed during the weighing in distilled water. When data is desired on the progressive changes occurring with increasing time

of immersion, the test specimen shall be immediately removed from the water after making this determination, dipped quickly into SDA-2B alcohol at room temperature, blotted lightly with filter paper to remove the water and alcohol, and replaced in the test liquid with the least possible lapse of time.

*(b) Specific Gravity Method for Water Soluble Liquids of Known Composition.*

—This method is not satisfactory for mixed liquids because the specific gravity of the absorbed liquid may differ from that of the bulk. The same procedure shall be used as for the water displacement method described in Paragraph (a), except that after blotting the test specimen with filter paper and weighing, it is not removed from the bottle nor weighed in water.

**Changes in Tensile Strength and Elongation**

10. (a) The tension tests as required in the following Paragraphs (b) to (d) shall be made in accordance with A.S.T.M. Methods D 412.

*(b) Original Properties.*—The original tensile strength and ultimate elongation shall be determined, using duplicate specimens to those which are immersed in liquid.

*(c) Immediate Deteriorated Properties.*—For determining the tensile strength and ultimate elongation immediately after removal from the liquid, the dumbbell-shaped specimens prescribed in Section 7 (c) shall be gaged and placed in test tubes as described in Section 6 (a) or (b) and 150 ml. of liquid added to each. If a nonvolatile liquid is used, the test tubes may then be placed in a thermostatically controlled air oven for the required length of time. However, if a volatile liquid is used, each test tube shall be fitted with a reflux condenser as described in Section 6 (a) and placed in a liquid bath thermostatically controlled at the desired temperature of

test. In either case a control test tube containing the test liquid or any other suitable medium and a precision thermometer shall be used along with the test tubes containing the test specimens. At the end of the required length of immersion time the specimens shall be removed from the test tubes and cooled immediately in a fresh sample of the same liquid at room temperature for not less than 30 min. nor more than 60 min. (This step is unnecessary if the immersion test is made at room temperature.) The test specimens shall then be dipped quickly in acetone, blotted lightly with filter paper, mounted in the testing machine, and tested immediately. The time interval between removal from the cold liquid and testing shall be not less than 2 nor more than 3 min.

*(d) Recovered Properties.*—For determining the tensile strength and ultimate elongation after recovery, the dumbbell-shaped specimens prescribed in Section 7 (c) shall be placed in test tubes as described in Section 6 (a) or (b) and 150 ml. of liquid added to each. The immersion shall be carried out as described in Section 8. At the end of the required length of immersion time, the specimens shall be removed from the test tube, suspended in air, protected from light and draft, and held at room temperature for  $4 \pm \frac{1}{4}$  hr., after which they shall be gaged and tested immediately.

**Calculations**

11. (a) The increase in weight (Section 8) shall be calculated as follows:

$$\text{Percentage increase in weight} = \frac{W_2 - W_1}{W_1} \times 100$$

where:

$W_1$  = initial weight, and

$W_2$  = weight after test.

(b) The increase in volume shall be calculated as follows:

*Water Displacement Method (Section 9 (a)):*

Percentage increase in volume =

$$\frac{(W_3 - W_4) - (W_1 - W_2)}{(W_1 - W_2)} \times 100$$

where:

$W_1$  = initial weight in air,

$W_2$  = initial weight in water,

$W_3$  = weight in air after the immersion test, and

$W_4$  = weight in water after the immersion test.

*Specific Gravity Method (Section 9 (b)):*

$$\text{Percentage increase in volume} = \frac{X \times Y}{Z}$$

where:

$X$  = percentage increase in weight,

$Y$  = specific gravity of sample, and

$Z$  = specific gravity of liquid.

(c) The tensile strength of the specimens directly after removal from the test liquid (Section 10 (c)) shall be calculated as follows:

$$T_1 = \frac{T_2}{\left(1 + \frac{V}{100}\right)^{\frac{2}{3}}}$$

where:

$T_1$  = tensile strength of composition in pounds per square inch, based on swollen area,

$T_2$  = tensile strength of swollen composition in pounds per square inch, based on original unswollen cross-sectional area, and

$V$  = percentage increase in volume (Section 11 (b)).

(d) The deterioration in tensile strength and ultimate elongation (Section 10) shown by the difference between the determinations before and after immersion shall be expressed as a percentage of the original property.

*Method B. Linear Measurement Method for Increase in Volume<sup>8</sup>*

## Apparatus

12. The apparatus shall consist of the following:

(a) *Immersion Tubes.*—Immersion tubes made from glass tubing having an outside diameter of 10 mm. and sealed at one end to an over-all length of approximately 250 mm. A glass rod 7.5 mm. in diameter and approximately 2 cm. in length, with square cut ends, shall be placed in the bottom of each tube. Small corks or rubber stoppers shall be provided for use in closing the tubes during the test when the vapor pressure of the liquid is not high. If the tubes are to be heated, a small breather hole shall be made in the stoppers so that they will not be blown out by the expanding air. Under conditions where the vapor pressure will be high, no stoppers shall be used but the tubes shall be cooled and sealed by means of a blast lamp after being filled. Identifying numbers may be placed on the corks or on tags wired to the tubes.

(b) *Racks for Immersion Tubes.*—Suitable racks for holding the tubes either vertically or at an angle to the horizontal just large enough to keep the liquid from flowing out if the cork comes out during the immersion period. In this way a large number of tubes may be placed in a small constant temperature oven or thermostat bath.

(c) *Reading Table.*—A reading table similar to that shown in Fig. 1. It may be constructed of any suitable material and shall have a glass top about 1 ft. square set at an angle of approximately 15 deg. to the horizontal. Standard millimeter graph paper, marked in ink at convenient lines to facilitate reading the increase in length of the test specimen, shall be fastened over the glass by means of thumb tacks or tape. A mirror shall be placed under the glass at an angle of about 45 deg. to the horizontal

<sup>8</sup> B. S. Garvey, Jr., "An Immersion Test for Rubber Compounds," ASTM BULLETIN, No. 109, March, 1941, p. 19.



and an ordinary electric light assembly fastened at the side as shown in Fig. 1, in order to reflect the light upward through the paper. This arrangement

by 1.6 mm. in width by 1.9 mm. in thickness, which shall be accurately cut from vulcanized sheets prepared as described in Sections 4 to 6 of A.S.T.M.

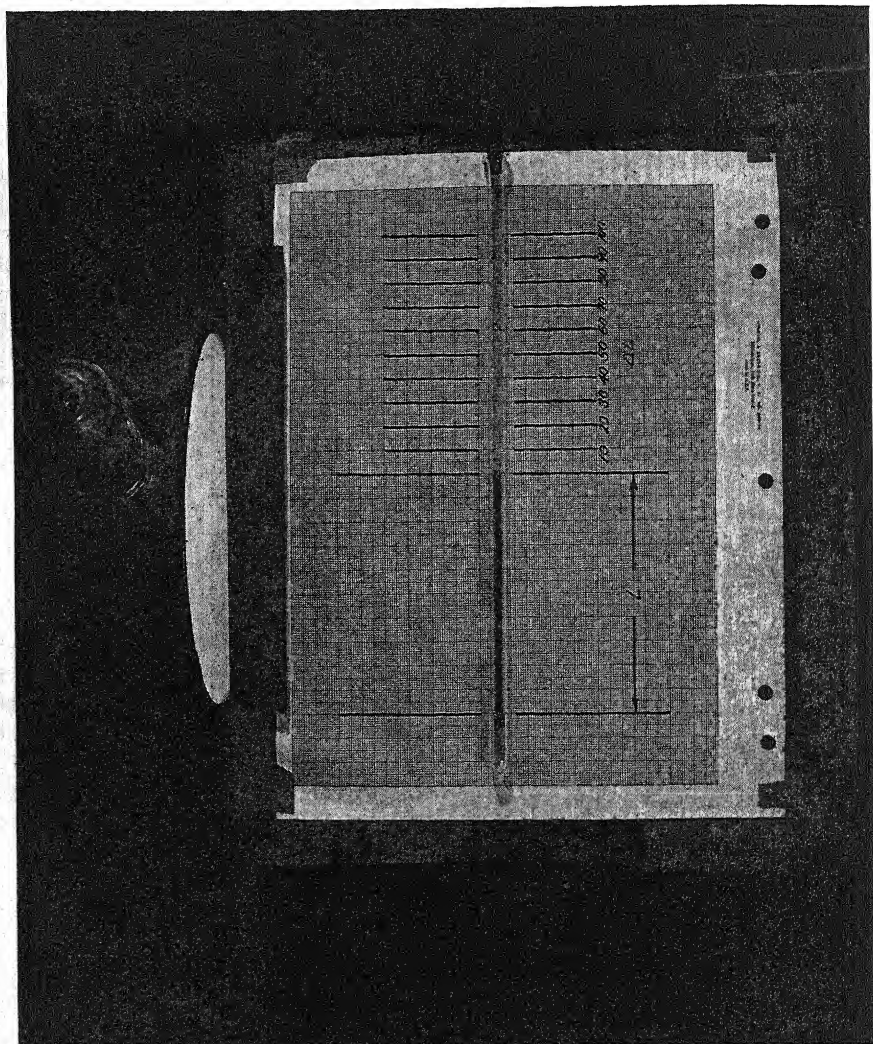


FIG. 1.—Reading Table.

prevents heating and consequent shrinkage of the paper.

#### Test Specimens

13. The standard test specimen shall be a rectangular strip 100 mm. in length

Methods D 15. One longitudinal and one transverse strip cut from each sheet at right angles to each other shall be tested and the results averaged so as to correct for possible grain effect. If specimens having thicknesses other

than standard or specimens cut in one direction only are tested, notation shall be made of such deviation from the standard and comparisons shall be made only between results from similar specimens.

the tube either corked or sealed depending on the type of liquid used. The tube shall be placed in the rack and kept at the specified temperature for the required length of time in a thermostatically controlled oven or bath, after which it shall

TABLE III.—RELATIONSHIP OF  $\Delta L$  TO  $\Delta V$ .

NOTE.— $\Delta L$  is the corrected difference in length of the test specimen and  $\Delta V$  is the corresponding change in volume expressed as a percentage of the original volume of the specimen.

$\Delta L$ , mm.	$\Delta V$ , per cent	$\Delta L$ , mm.	$\Delta V$ , per cent	$\Delta L$ , mm.	$\Delta V$ , per cent	$\Delta L$ , mm.	$\Delta V$ , per cent	$\Delta L$ , mm.	$\Delta V$ , per cent
0.5	1.5	20.5	75.0	40.5	177.3	60.5	313.5	80.5	488.1
1.0	3.0	21.0	77.2	41.0	180.3	61	317.3	81	493.0
1.5	4.5	21.5	79.4	41.5	183.3	61.5	321.2	81.5	498.0
2.0	6.1	22.0	81.6	42.0	186.3	62	325.2	82	502.9
2.5	7.6	22.5	83.8	42.5	189.3	62.5	329.1	82.5	507.9
3.0	9.2	23.0	86.1	43.0	192.4	63	333.1	83	512.8
3.5	10.8	23.5	88.4	43.5	195.5	63.5	337.1	83.5	517.9
4.0	12.5	24.0	90.7	44.0	198.6	64	341.1	84	523.0
4.5	14.1	24.5	93.0	44.5	201.7	64.5	345.1	84.5	528.1
5.0	15.8	25.0	95.3	45.0	204.9	64	349.2	85	533.2
5.5	17.4	25.5	97.5	45.5	208.0	65.5	353.3	85.5	538.3
6.0	19.1	26.0	100	46.0	211.2	66	357.4	86	543.5
6.5	20.8	26.5	102.4	46.5	214.4	66.5	361.6	86.5	548.7
7.0	22.5	27.0	104.8	47.0	217.7	67	365.7	87	553.9
7.5	24.2	27.5	107.2	47.5	220.9	67.5	369.9	87.5	559.2
8.0	26.0	28.0	109.7	48.0	224.2	68	374.2	88	564.5
8.5	27.7	28.5	112.2	48.5	227.3	68.5	378.5	88.5	569.8
9.0	29.5	29.0	114.7	49.0	230.8	69	382.7	89	575.1
9.5	31.3	29.5	117.2	49.5	234.1	69.5	387.0	89.5	580.5
10.0	33.1	30.0	119.7	50.0	237.5	70	391.3	90	585.9
10.5	34.9	30.5	122.2	50.5	240.9	70.5	395.6	90.5	591.3
11.0	36.8	31.0	124.8	51.0	244.3	71	400.0	91	596.8
11.5	38.6	31.5	127.0	51.5	247.8	71.5	404.4	91.5	602.3
12.0	40.5	32.0	130.0	52	251.2	72	408.8	92	607.8
12.5	42.4	32.5	132.6	52.5	254.7	72.5	413.3	92.5	613.3
13.0	44.3	33.0	135.3	53	258.2	73	417.8	93	618.9
13.5	46.1	33.5	137.9	53.5	261.7	73.5	422.3	93.5	624.5
14.0	48.2	34.0	140.6	54	265.2	74	426.8	94	630.1
14.5	50.0	34.5	143.3	54.5	268.8	74.5	431.3	94.5	635.8
15.0	52.1	35.0	146.0	55	272.4	75	435.9	95	641.5
15.5	54.1	35.5	148.7	55.5	276.0	75.5	440.1	95.5	647.2
16.0	56.1	36.0	151.5	56	279.6	76	445.2	96	653.0
16.5	58.1	36.5	154.3	56.5	283.3	76.5	449.8	96.5	658.8
17.0	60.2	37.0	157.1	57	287.0	77	454.5	97	664.5
17.5	62.2	37.5	159.9	57.5	290.7	77.5	461.2	97.5	670.3
18.0	64.3	38.0	162.8	58	294.4	78	464.0	98	676.2
18.5	66.4	38.5	165.7	58.5	298.2	78.5	468.8	98.5	682.1
19.0	68.5	39.0	168.6	59	302.0	79	473.5	99	688.1
19.5	70.6	39.5	171.5	59.5	305.8	79.5	478.3	99.5	694.1
20.0	72.8	40.0	174.4	60	309.6	80	483.2	100	700.0

## Procedure

14. (a) Each test specimen shall be placed in an immersion tube with the lower end resting on the glass rod at the sealed end of the tube. The original length of the specimen shall then be checked using the reading table. Any deviation from the standard length of 100 mm. shall be noted for use in correcting the subsequent change in length after immersion. Then, 10 ml. of the immersion liquid shall be added and

be removed and allowed to cool to room temperature. The difference in length of the specimen from the original 100 mm. shall then be read to the nearest 0.5 mm. by means of the reading table shown in Fig. 1 and corrected for any deviation from the standard length that may have been noted.

(b) In making the reading, parallax is eliminated when the line being read goes straight across the tube as seen through the liquid. Two black dots

will be observed in the wall of the tube and these should appear to coincide with the corresponding line under the tube.

(c) Using the corrected difference in length ( $\Delta L$ ), the corresponding change in volume ( $\Delta V$ ) expressed as a percentage of the original volume shall be read from Table III. The values for both  $\Delta L$  and  $\Delta V$  shall be recorded. The average  $\Delta V$  for the longitudinal and transverse specimens from a given sample shall be taken as the increase in volume. When information is desired on the rate of swelling, readings may be made without removing the specimen from the tube after successive periods at the specified temperature. In some cases, the test specimen curls slightly during immersion causing an error of as much as 0.5 mm. in  $\Delta L$ . This may be prevented by

placing in the tube a piece of stiff wire bent zigzag in a plane so as to hold the specimen flat.

#### Report

15. The report shall include the following:

- (1) The results calculated in accordance with Section 11,
- (2) All observed and recorded data,
- (3) The exposure period,
- (4) The temperature of exposure,
- (5) The liquid used,
- (6) Statement of condition of exposed specimens from visual and manual examination,
- (7) The duration, temperature, and date of vulcanization of the test specimens, and
- (8) The dates of the various periods of exposures.

# *Tentative Method of Test for*

## PERMEABILITY OF VULCANIZED RUBBER OR SYNTHETIC ELASTOMERS TO VOLATILE LIQUIDS<sup>1</sup>



A.S.T.M. Designation: D 814 - 46 T

ISSUED, 1944; REVISED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is intended for use in evaluating the permeability of vulcanized rubber or synthetic elastomers with respect to volatile liquids diffusing into air. It is applicable only to the materials in sheet form of moderate thickness and is principally useful for comparing the relative permeability of different materials to the same liquid or the diffusion of different liquids through the same material. Comparisons should not be made between determinations in which both different rubbers and different liquids are used.

### Apparatus

2. (a) The test apparatus shall consist essentially of glass permeability jars (Paragraph (b)) for each liquid on which the test specimens are mounted, and a suitable rack for supporting the jars in an inverted position in such a way as to allow free circulation of air over the

surface of the specimen. The apparatus shall be kept in a constant temperature cabinet or room at  $25 \pm 2.8$  C

(b) *Permeability Jar*.—The glass permeability jar shall be of approximately  $\frac{1}{2}$ -pt. capacity with an opening of  $2.375 \pm 0.015$  in. in inside diameter and a wall  $\frac{1}{8}$  in. in thickness at the opening which shall be ground flat without rounded edges to a smooth ground-glass finish at the contact surface. The opening shall be equipped with a metal screw ring clamp holding a smooth-edged, flat, sheet-metal ring  $2.188 \pm 0.015$  in. in inside diameter and approximately  $2\frac{1}{4}$  in. in outside diameter. The ring clamp and ring serve as a clamping device for holding the specimen against the ground-glass edge of the jar with a circular specimen area  $2\frac{3}{16}$  in. in diameter exposed to the air while permitting the screw clamp to be tightened to form a leak-proof seal without subjecting the specimen to torsional strain. When testing extensible materials, a circular disk of 16 to 20-mesh screen shall be placed inside the screw ring clamp between it and the flat metal ring to prevent stretching of

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

the specimen during the test. The screen disk may be soldered in place on top of the sheet metal ring if desired.

NOTE.—A suitable permeability jar may be prepared from a 1/2-pt. Mason jar equipped with a Kerr type screw cap provided the edge of the jar is properly ground and finished with fine emery and the cap is cut with smooth flattened edges to form the required circular opening.

### Test Specimens

3. (a) The test specimens shall consist of circular disks  $2\frac{1}{8}$  in. in diameter, cut with a sharp-edged die from a sheet of the material being tested. The thickness of the sheet which shall not exceed 0.125 in. shall be measured using a dial micrometer graduated to 0.001 in. having a presser foot  $0.25 \pm 0.01$  in. in diameter exerting a total force of  $3.0 \pm 0.1$  oz.

(b) For evaluating the permeability of a particular material with respect to a given fluid, three specimens having the same nominal thickness within a tolerance of plus or minus 3 per cent shall be tested. The permeability of the material shall be taken as the average of the values obtained from the two specimens giving the lowest results providing these check within plus or minus 20 per cent of the average so determined.

### Procedure

4. (a) With the permeability jar in an upright position, 200 ml. of the test liquid shall be placed in it after which the test specimen shall be fitted over the mouth of the jar, the ring and wire screen placed in position and the screw ring clamp screwed down lightly. The assembly shall then be held in the constant temperature cabinet or room at  $25 \pm 2.8$  C. with the test disk lightly in place until temperature equilibrium is obtained when the ring shall be screwed down tightly and the assembly allowed to stand in an upright position for 2 hr. A tare assembly shall be made using a

duplicate jar and a test specimen of the same size but without the addition of the test liquid. Suitable weighting material such as lead shot shall be placed in the tare jar to bring its weight to approximately that of the assembled jar and contents. The tare jar shall be conditioned at the test temperature, sealed, and handled throughout the test in a manner similar to that used for the test jar. The test jar shall then be placed on a balance of suitable capacity capable of weighing to 0.005 g. and counterbalanced against the tare jar adding weights as required for equilibrium. The original weight of the test assembly shall be taken as that of the tare corrected for the added weights and shall be recorded to the nearest 0.005 g.

(b) After weighing, each test jar assembly shall be inverted and placed on the rack, supported in such a way that the entire inner surface of the specimen remains in contact with the liquid throughout the test. If the rack is located in a conditioning cabinet care shall be taken to remove diffused vapors from the chamber and to assure free circulation of fresh air by means of a fan or other suitable device. Twenty-four hours after inverting the jar, it shall be again weighed against the tare jar. Any excessive loss of weight indicates that leakage due to improper seal is occurring and such test assemblies shall be removed and replaced by new assemblies properly prepared. The test assembly shall be returned to the rack and reweighed against the tare after periods of  $120 \pm 2$  hr. and  $192 \pm 2$  hr. from the initial inversion. The difference in weight between those obtained at 120 hr. (5 days) and at 192 hr. (8 days) divided by 3 shall be considered as the average weight of fluid lost per 24 hr.

NOTE.—In this test, the specimens are allowed to remain in contact with the fluid for 5 days before starting measurement of the loss in order to permit the rate of diffusion of the fluid to become uniform.

**Calculation and Report**

5. (a) *Calculation.*—The permeability of each specimen with respect to the test liquid employed shall be expressed as fluid ounces of liquid lost per 24 hr. per square foot of wetted area of the specimen. It shall be calculated as follows:

$$\text{Permeability, fluid oz. per 24 hr. per sq. ft.} = \frac{1.099 \times \text{average wt. in grams of fluid lost per 24 hr.}}{\text{sp. gr. of fluid}}$$

(b) *Report.*—The report shall include the following:

- (1) Identification of the test specimen.
- (2) The thickness of the test specimen to the nearest 0.001 in.
- (3) Identification of the test liquid.
- (4) The permeability of the test specimen to the test liquid expressed in fluid ounces per 24 hr. per sq. ft. of wetted area.

# *Tentative Method of Test for*

## LOW-TEMPERATURE BRITTLINESS OF RUBBER AND RUBBER-LIKE MATERIALS<sup>1</sup>



A.S.T.M. Designation: D 736 - 46 T

ISSUED, 1943; REVISED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is intended to determine the ability of compounds made from rubber or rubber-like materials to resist the effect of low temperatures in causing them to become brittle so as to fracture or crack when bent. Standard specimens are exposed to specified low temperatures for definite periods after which the specimens are bent in a prescribed manner and any fracture or cracking noted. The procedure is commonly called the "Thiokol" method.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Cold Chamber*.—The cold chamber in which the test specimens are exposed to the low temperature shall be of sufficient size to contain the flexing fixture when loaded with specimens, and so arranged as to permit the operation of the fixture to bend the specimens with-

out removal from the chamber. It shall be capable of maintaining within it a uniform atmosphere of cold, dry air or a mixture of air and carbon dioxide at specified temperatures within a tolerance of plus or minus 2 F. Standard test temperatures shall be -40 F. and -70 F.

(b) *Flexing Fixture*.—The flexing fixture shall consist of two parallel plates each having a width of at least 2 in. so supported in guides that they may be rapidly moved from a position  $2\frac{1}{2}$  in. apart until they are separated by a distance of 1 in. Suitable clamping bars or devices shall be provided for holding the ends of the specimens for a distance of  $\frac{1}{2}$  in. at the corresponding edge of each plate so that when mounted, the specimens form similar bent loops between the plates. A satisfactory flexing fixture is shown in Fig. 1.

### Test Specimens

3. (a) The test specimens shall conform in shape to die C as specified in the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.



Designation: D 412)<sup>3</sup> and shall have a thickness of  $0.080 \pm 0.010$  in.

(b) At least two specimens from each compound shall be tested.

#### Procedure

4. The test specimens shall be mounted in loop position between the plates of the flexing fixture with the enlarged ends spaced at least  $\frac{1}{8}$  in. apart and held in the clamps for a dis-

mination of the exposure period and while still in the cold chamber, the plates of the flexing fixture shall be moved as rapidly as possible from the  $2\frac{1}{2}$  in. distance of separation to a position where they are 1 in. apart. The specimens shall then be examined for fracture or visible cracks.

#### Results

5. (a) When two specimens are tested

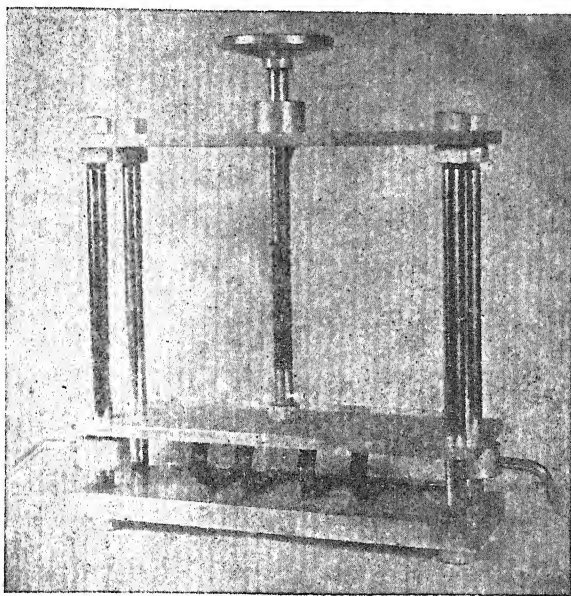


FIG. 1.—Flexing Fixture for Low-Temperature Brittleness Test.

tance of  $\frac{1}{4}$  in. With the plates in the open position separated  $2\frac{1}{2}$  in., the fixture containing the specimens shall be placed in the cold chamber and exposed for the specified period to cold, dry air or a mixture of air and carbon dioxide at the specified temperature. Standard exposure periods shall be 5 hr. for compounds made from synthetic rubber or rubber-like materials and 96 hr. for natural rubber compounds. At the ter-

and neither one fractures nor shows cracks after being tested, the compound shall be considered as having passed the brittleness test. If both specimens crack, the compound shall be considered to have failed.

(b) If only one specimen fractures or cracks, the result is inconclusive and two additional specimens shall be tested. If either one of these cracks, the compound shall then be considered to have failed.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

**Report**

6. The report shall include the following:

(1) The results of the test expressed as "passed" or "failed,"

(2) The temperature of the cold chamber,

(3) The duration of the exposure period,

(4) Identification of the material tested including description of any special treatment prior to test, and

(5) Date of manufacture of the material, if known, and date of test.

*Tentative Recommended Practice for*  
CONDITIONING OF RUBBER AND PLASTIC MATERIALS  
FOR LOW-TEMPERATURE TESTING<sup>1</sup>



A.S.T.M. Designation: D 832 - 46 T

ISSUED, 1945; REVISED, 1946.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Scope**

1. (a) This recommended practice covers the general theory underlying all low-temperature testing of rubber and plastic materials (Note). It is designed to acquaint persons making mechanical tests at low temperatures with the characteristic behavior of natural rubber, synthetic rubber, rubber-like materials, and plastics, and to outline the conditioning procedure necessary for making these tests within the limits of present knowledge.

(b) This recommended practice also covers the various types of low-temperature cabinets or "cold boxes," as they are commonly called, which are in general use for both conditioning and testing of rubber and plastic materials at sub-zero temperatures. In addition to a description of the various types of commercial equipment, details are given of a low-cost low-temperature cabinet

which can be made in the laboratory from inexpensive and readily available materials.

NOTE.—This recommended practice is intended to apply particularly to the following methods of the American Society for Testing Materials:

Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736),<sup>3</sup>

Tentative Method of Test for Brittle Temperature of Plastics and Elastomers (A.S.T.M. Designation: D 746)<sup>3</sup>

Tentative Method of Test for Stiffness in Flexure of Nonrigid Plastics (A.S.T.M. Designation: D 747),<sup>3</sup>

Standard Method of Test for Young's Modulus in Flexure of Natural and Synthetic Elastomers at Normal and Subnormal Temperatures (A.S.T.M. Designation: D 797),<sup>3</sup> and

Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3,4</sup>

**General Considerations**

2. Regardless of the low-temperature test employed, one of the first stages

<sup>1</sup> Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> For additional information relating to this method of test, see F. L. Yerzley and D. F. Fraser, "Effects of Low Temperatures on Neoprene Vulcanizates," *Industrial and*

in establishing a satisfactory technique is the specification of the time and temperature of exposure of the test specimen. It has been demonstrated

### Simple Temperature Effects

3. Most of the elastic properties of rubber-like materials undergo change as the temperature is reduced. At

TABLE I.—DIFFERENTIATION BETWEEN CRYSTALLIZATION AND SECOND-ORDER TRANSITION.

Property	Crystallization	Second-Order Transition
Physical effects (6, 7, 9, 11) <sup>a</sup>	Becomes stiff (hard) but not necessarily brittle.	Becomes stiff and brittle.
Temperature - volume relation (1, 4, 5, 7)	Significant change in volume.	No change in volume, but definite change in coefficient of thermal expansion.
Latent heat effect (1, 2, 3, 4, 8, 10)	Heat evolved on crystallization.	No heat effect, but definite change in specific heat.
Rate (1, 2, 6, 9, 11)	Hours or days required. Dependent on temperature. In general, as temperature is lowered rate of crystallization increases to maximum and then decreases. Time is important. Rate also varies with composition and state of cure.	Rapid; takes place within a definite narrow temperature range regardless of thermal history of specimen. Time is not important unless plasticizers are present.
Temperature of occurrence (1, 2, 3, 4, 9, 10, 11)	Probably not below -50 C. Maximum rate probably not below -30 C. Optimum temperature is specific to base polymer.	Very wide limits, depending on composition.
Effect on molecular structure (1)	Orientation of molecules similar to crystallization of simpler substances.	Change in type of motion of segments of molecule.
Materials exhibiting properties (1, 5, 6, 7, 9, 11)	Unstretched polymers as follows: natural rubber (low-sulfur vulcanizates), Neoprene (excepting type FR), Thiokols A and N, vinylidene chloride, polyethylene, nylon plastics. The following materials do not crystallize: GR-I (excepting when stretched), GR-A, GR-S.	All rubber-like materials and plastics.

<sup>a</sup> The boldface numbers in parentheses refer to the following references:

- (1) Norman Bekkedahl, "Forms of Rubber as Indicated by Temperature-Volume Relationships," *Journal of Research, Nat. Bureau Standards*, Vol. 13, No. 3, September, 1934, pp. 411-431. (*Research Paper RP 717*.)
- (2) Norman Bekkedahl and L. A. Wood, "Influence of the Temperature of Crystallization on the Melting of Crystalline Rubber," *Journal of Chemical Physics*, Vol. 9, No. 2, February, 1941, p. 193.
- (3) Norman Bekkedahl and L. A. Wood, "Crystallization of Vulcanized Rubber," *Industrial and Engineering Chemistry*, Vol. 33, March, 1941, p. 381.
- (4) Norman Bekkedahl and Russell B. Scott, "Specific Heat of the Synthetic Rubber Hycar OR from 15° to 340° K.," *Journal of Research, Nat. Bureau Standards*, Vol. 29, No. 1, July, 1942, pp. 87-95. (*Research Paper RP 1487*.)
- (5) R. F. Boyer and K. S. Spencer, "Thermal Expansion and Second-Order Transition Effects in High Polymers. Part I. Experimental Results," *Journal of Applied Physics*, Vol. 15, p. 398 (1944).
- (6) R. F. Clash and R. M. Berg, "Stiffness and Brittleness Properties of Nonrigid Vinyl Chloride-Acetate Resin Compounds," Symposium on Plastics, p. 54, Philadelphia District Meeting, Am. Soc. Testing Mats. (1944). (Symposium issued as separate publication.)
- (7) R. F. Clash and L. M. Rynkiewicz, "Thermal Expansion Properties of Plastic Materials," *Industrial and Engineering Chemistry*, Vol. 36, p. 279 (1944).
- (8) D. B. Forman, "Effect of Elastomer Crystallization on Tests for Freeze Resistance," *Industrial and Engineering Chemistry*, Vol. 36, p. 738 (1944).
- (9) J. W. Liska and F. S. Conant, "Some Low Temperature Properties of Elastomers," *Journal of Applied Physics*, Vol. 15, p. 767 (1944).
- (10) J. W. Liska, "Effect of Low Temperatures on Young's Modulus of Elastomers," *Industrial and Engineering Chemistry*, Vol. 36, p. 40 (1944).
- (11) R. D. Rands, W. J. Ferguson, and J. L. Prather, "Specific Heat and Increases of Entropy and Enthalpy of the Synthetic Rubber GR-S from 0° to 330° K.," *Journal of Research, Nat. Bureau Standards*, Vol. 33, No. 1, July, 1944, p. 63. (*Research Paper RP 1596*.)

that the following distinct changes take place on lowering the temperature:

- Simple temperature effects,
- Second-order transitions (vitrification),
- Crystallization (first-order transitions), and
- Effects associated with plasticizers.

temperatures above those at which transitions of the second order take place, these changes are usually manifested by increased "logginess" of the specimen, loss in resilience, increase in modulus, and increase in hardness. It should be noted that as the temperature is decreased, resilience will decrease

to a minimum and then increase with further decline in temperature. This progressive increase in resilience with declining temperature continues until some temperature below the second-order transition is reached. Changes due to simple temperature effects are complete when the test specimens have reached thermal equilibrium. The magnitude of these changes depends on the composition of the material and the test temperature.

### Second-Order Transitions (Vitrification)

4. Transitions of the second order, which are exhibited by all plastics and all rubber-like materials, do not depend critically on time. They take place quickly within a definite temperature range of a few degrees. Considerable change in elastic properties usually precedes a second-order transition. Measurement of these effects should be limited to exposure times required for thermal equilibrium, otherwise crystallization or plasticizer - time effects may influence the results. A convenient differentiation between crystallization and second-order transitions is provided by the summary given in Table I.

### Crystallization (First-Order Transitions)

5. Changes in properties resulting from crystallization may take hours, days, weeks, or longer, depending on the material being tested and the particular temperature employed. Crystallization effects begin some time after thermal equilibrium has been established and are manifested by increase in hardness or modulus. Only a relatively small number of materials, such as natural rubber, Thiokol A, Neoprene GR-M, Neoprene CG, Neoprene E, polyethylene,<sup>6</sup> and nylon, exhibit crystallization. These

materials conform to the present concept that only linear polymers made from a single monomer are crystallizable. The temperature at which natural rubber crystallizes most rapidly is about  $-25^{\circ}\text{C}$ . and for Neoprene GR-M it is reported to be about  $0^{\circ}\text{C}$ . Both above and below these temperatures crystallization proceeds at a slower rate. Accordingly, any attempt to compare materials (particularly those subject to change in properties resulting from crystallization or plasticizer - time effects) on a basis of exposure at a given temperature for a specific time is almost certain to be misleading. Such a specific temperature may be near the optimum rate of crystallization of one of the materials and many degrees above or below the optimum of another. A convenient differentiation between crystallization and second-order transitions is provided by the summary given in Table I.

### Effects Associated With Plasticizers

6. When the material being tested contains considerable amounts of certain plasticizers, time effects not necessarily associated with crystallization may be observed. These effects occur over a wide range of time, temperature, and composition. Some of these effects may be explained on the basis of limited low-temperature solubility of such plasticizers in the compound. If the original plasticizer concentration is less than the amount corresponding to saturation at the test temperature, no time effects will be observed. The effects consist of delayed stiffening and, in some instances, an elevation of the brittle temperature. In the case of elevation of the brittle temperature, plasticized compositions may become brittle after an extended exposure to temperatures slightly higher than their brittle temperatures. This effect appears to be limited to a very narrow temperature range, which is in

<sup>6</sup> Polyethylene may crystallize in a very short time—in seconds or minutes at  $105$  to  $112^{\circ}\text{C}$ .

contrast to the relatively wide range in which delayed stiffening may occur.

#### CONDITIONING PROCEDURES FOR MECHANICAL TESTS

##### Tests for Effects of Second-Order Transition (Vitrification) Only

7. The test specimen shall be held at each test temperature until it reaches a uniform temperature. For test specimens 0.25 in. or less in thickness, the recommended conditioning time in air is 20 min. and in liquid is 10 min. Thicker test specimens, as well as some compositions, will require longer conditioning times. When the equilibrium temperature has been reached, the required measurements of modulus, hardness, or brittleness should be made immediately, while maintaining the specimen at the same temperature.

##### Tests for Effects of Crystallization (First- Order Transition) Only

8. These tests shall be conducted at the temperatures of maximum rate of crystallization (for example,  $-25^{\circ}\text{C}$ . for natural rubber and  $0^{\circ}\text{C}$ . for Neoprene GN. The temperature of the test specimen should be allowed to come to equilibrium as described in Section 7. When the equilibrium temperature has been reached, one set of the required modulus or hardness measurements should be made immediately. The test specimen should be aged for 72 hr. at the specified temperature, and a second set of measurements comparable to the first should then be made. Increased stiffness is an indication of crystallization or a plasticizer effect.

##### Tests for the Effects Associated with Plasticizers

9. The optimum temperature for determining the increased stiffness caused by the time effects of plasticizers has not been determined, but

data are being accumulated with this end in view. It is suggested that tests should be conducted at temperatures  $5^{\circ}\text{C}$ . higher than the brittle temperature. The procedure described in Section 8 should be followed, except that in measurements for brittleness that may occur after an extended exposure to a given temperature, a slight modification of the procedure described in Section 8 is suggested. In this case the temperature should also be  $5^{\circ}\text{C}$ . higher than the observed brittle point (see Section 7). Tests for brittleness should be made after periods of 15 and 60 min. exposure in air followed by as many additional tests as desired through a 72-hr. exposure period. Long exposure in a liquid medium may give erroneous results.

#### LOW-TEMPERATURE CABINETS<sup>6</sup>

##### General Requirements

10. (a) The low-temperature cabinets may be of the mechanically refrigerated type, dry ice type, or the so-called "packaged air" type, but shall conform to the following requirements:

(1) The heat-transfer medium in the test chamber shall be air or a mixture of air and carbon dioxide.

(2) The temperature variation throughout the effective test chamber shall be within  $\pm 2^{\circ}\text{F}$ . of the specified testing temperature.

(3) The heat-transfer medium shall be thoroughly circulated in the test chamber by means of mechanical agitation. A fan, suitably located in the test specimen chamber, can be used for this purpose.

(4) Automatic temperature control

<sup>6</sup> Satisfactory low-temperature cabinets have been supplied by the following manufacturers:

Bowser Co., Inc., Refrigeration Div., Terryville, Conn.

American Instrument Co., 8010-8020 Georgia Ave., Silver Spring, Md.

Precision Scientific Co., 1750 W. Springfield Ave., Chicago 47, Ill.

Tenney Engineering, Inc., 40 Farrand St., Montclair, N. J.

shall be used. A bimetallic thermostat has been found to be satisfactory.

(b) The size of the test chamber is optional. However, an 8-cu. ft. compartment 24 by 24 by 24 in. is sufficient for all of the low-temperature tests listed in Section 1. For tests such as A.S.T.M. Method D 736, a 1-cu. ft. test chamber may be used. The equipment for A.S.T.M. Method D 747 is frequently installed in a "walk in" type of cold room when it is available.

(c) The door can be located either in the top of the test cabinet or in the side. A side-opening door is convenient and is necessary where the equipment involved must be operated from the side. However, it has the disadvantage of allowing the cold air to pour out when the door is opened. In general, a window in the door is desirable for observation and the reading of test equipment dials. This window should be, preferably, of the 5-ply Thermopane construction, or its equivalent, to prevent frosting.

(d) For units that are in continuous use, a 6-in. wall of ground cork, fiberglass, rock wool, or other suitable insulation will be found desirable in reducing thermal losses and in conservation of dry ice or refrigeration. For the operation of the equipment involved in A.S.T.M. Method D 676, handholes equipped with insulated sleeves are frequently installed in the walls of the test cabinet.

#### Mechanically Refrigerated Units

11. (a) In general, the mechanically refrigerated low-temperature cabinets consist of a multiple-stage compressor and suitable cooling coils which surround the test chamber. Suitable insulation is provided between the test chamber and the outside walls of the cabinet. Automatic temperature control is obtained by either a thermostatic control located

in the test chamber, which turns the compressor on or off, or by a suitable pressure control, which regulates the refrigerant temperature. Air is used as the heat-transfer medium in the test specimen compartment.

(b) This type of equipment is well adapted to continuous operation at a fixed temperature. Except for rather high maintenance and initial costs, this type of equipment is less expensive to operate continuously from a power standpoint than the dry ice units. Temperatures as low as  $-90^{\circ}\text{F}$ . can be obtained with commercial units. By the installation of electric strip heaters and suitable automatic controls, temperatures up to room temperature and above can be obtained.

#### Dry Ice Units (Direct Type)

12. (a) In the direct type of dry ice low-temperature cabinets, a suitable fan or blower, located in the dry ice compartment, circulates the carbon dioxide vapor from the dry ice chamber into the test specimen chamber and back. By means of a preset damper between the dry ice compartment and the test chamber, the inlet and outlet openings can be adjusted for maximum efficiency. A bimetallic thermostat, located in the test compartment, controls the "on" and "off" operation of the fan in the dry ice compartment, thus providing automatic temperature control. To insure uniformity of temperature in the test compartment, a fan is provided.

(b) Commercial units are available in either 1- or 8-cu. ft. capacities and provide a temperature range of from  $-100^{\circ}\text{F}$ . to  $+220^{\circ}\text{F}$ . These units are particularly adapted for intermittent use where a wide range of testing temperatures is desired. Only a short time is required to cool the test chamber to a low temperature.



**Dry Ice Units (Indirect Type)**

13. (a) In the indirect type of dry ice low-temperature cabinets, air is used as the heat-transfer medium and no carbon dioxide from the dry ice comes in contact with the test specimens. The test chamber is cooled by circulating the carbon dioxide vapor completely around the

(b) Commercial units are available in either a 1- or 8-cu. ft. test chamber capacity. These units operate over a temperature range of  $-90$  to  $+220$  F. Two fans are provided, one for circulating the carbon dioxide around the outside of the test chamber and the other for circulating the air heat transfer medium

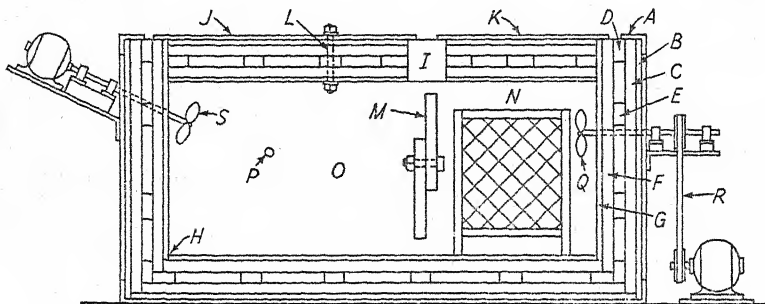


FIG. 1.—Schematic Diagram of Low-Temperature Test Cabinet.

- A—Angle iron frame of the desired dimensions, constructed from 1  $\frac{1}{2}$  or 2-in. angles with welding at all corners.
  - B—Layer of  $\frac{1}{4}$ -in. hard structural board.
  - C—Layer of  $\frac{1}{2}$ -in. thermal insulating board.
  - D— $\frac{1}{2}$  by 2-in. strips of thermal insulating board, glued in place.
  - E— $\frac{1}{2}$  by  $\frac{1}{4}$ -in. blocks of thermal insulation, glued in place at intervals of 10 or 12 in. to provide an air space.
  - F—Layer of  $\frac{1}{2}$ -in. thermal insulating board.
  - G—Layer of  $\frac{1}{4}$ -in. hard structural board.
  - H—Gummied paper in the corners and on the top edges (not shown in the sketch). No other bracing of the panels is needed because all but the last layers are held by interlocking at the corners, and the gummied paper serves very well to hold the inner panels and to finish corners.
  - I—Wood support between lids.
  - J and K—Lids of the same general construction, except that lid J is bolted together.
  - L—Bolts consisting of  $\frac{1}{2}$ -in. wood dowel pins, threaded at both ends and spaced every 10 or 12 in. Washers and nuts are of steel.
  - M—Partition arranged so that the top and bottom openings are adjustable.
  - N—Dry ice compartment with a basket of expanded metal to hold the dry ice.
  - O—Test chamber.
  - P—Thermostat element. This may be of the bi-metal type except where exposed to vibration which may cause the contacts to chatter. A relay is advisable with this type to protect the points. Thermocouple control is more expensive but is satisfactory under all conditions.
  - Q—Fan for circulating heat-transfer medium over the dry ice.
  - R—V-Belt drive, selected to run the fan Q because of the ease of changing pulleys to change fan capacity to the correct amount. It is necessary to avoid over or under amounts of blowing to control the temperature without leakage at the lids. Leakage, of course, uses excess ice and permits the entry of moisture. For these reasons it is preferred to use intermittent operations of the fan Q, rather than automatic control of openings in partition M. Automatic operation of Louvers in partition M would be satisfactory if made to close when the drive is shut off and open wide when the drive is running. Some control of partition M is necessary for relatively high temperatures in chamber O. Otherwise, convection and conduction through the partition may keep test chamber O colder than desired.
  - S—Circulating fan which runs continuously to prevent high and low temperature strata in the test chamber.
- Alternate Construction.*—Dry ice compartment N and test chamber O may be made in separate boxes if desired, connected by two flexible hose lines. Blower Q is then placed in one of the hose lines.

outside of the test chamber which, in turn, is insulated from the outside of the cold box. In general, this type is of a more costly construction than the direct type and is not quite as efficient. The time required to cool the test chamber to a low temperature is somewhat greater and is more comparable in this respect to the mechanically refrigerated units.

inside the test chamber. An automatic switch on the door shuts down the latter fan when the door is opened, thus tending to prevent frosting.

**Packaged Air Units**

14. (a) It is frequently desirable to enclose a piece of test equipment in a separate test chamber and circulate temperature-regulated cold air or carbon

dioxide from a separate unit<sup>5</sup> by means of insulated ducts or pipes. This type of unit is somewhat similar to the direct type of dry ice unit (Section 14) but contains an auxiliary blower to force the cold air through the pipes to the separate test chamber. It has the advantage of being portable and can be connected to different pieces of equipment.

(b) In some cases the temperature is controlled by a Modutrol motor-driven damper, or its equivalent, which bypasses the cold chamber or specimen chamber rather than a control of the fan motor itself. Motor or solenoid-driven dampers which operate in the dry ice compartment for the control of temperature may cause trouble due to frosting of the mechanism. No method which is entirely satisfactory has been devised to date to remove moisture from the heat transfer medium and completely eliminate frosting. Desiccants such as calcium chloride and calcium sulfate have been used.

#### Laboratory-Built Units

15. (a) In Fig. 1 is shown a schematic diagram of the essential requirements for an inexpensive dry ice low-temperature cabinet of the direct type, which can be built in any laboratory from mate-

rials which are readily available. The cabinet is made of hard structural board and thermal insulating board, with a central dead air space for insulation. The unit is divided into two compartments by the adjustable dampers. The size of the inlet and outlet openings can be adjusted for maximum efficiency by means of these dampers. The fan in the test compartment insures positive circulation and resultant uniformity of temperature in this chamber. Automatic temperature control to  $-90^{\circ}\text{F}$ . is obtained by means of a bimetallic thermoregulator which turns the fan in the dry ice chamber on and off. By the use of electric strip heaters in place of dry ice, the unit will function equally well at elevated temperatures.

(b) This unit can be built to any convenient size and can be provided with either top or side opening door, with or without a window. The wall thickness, while not great, is sufficient for good temperature regulation, and dry ice consumption is not excessive. Where continuous operation is contemplated and dry ice consumption is a major factor, a 6-in. wall of ground cork, fiber glass, rock wool or other suitable insulation can be used. Where walls are perforated for fixture attachment, bakelite or fiber tubes should be used.

# *Tentative Specifications for*

## RUBBER AND SYNTHETIC RUBBER COMPOUNDS FOR AUTOMOTIVE AND AERONAUTICAL APPLICATIONS<sup>1</sup>



A.S.T.M. Designation: D 735 - 46 T

ISSUED, 1943; REVISED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures, for use pending adoption as standard. Suggestions for revision should be addressed to the Headquarters of the A.S.T.M., 1916 Race St., Philadelphia 3, Pa., or of the S.A.E., 29 W. 39th. St., New York 18, N. Y.

*These specifications were prepared jointly by the Society of Automotive Engineers and the American Society for Testing Materials.*

### Scope

1. (a) These specifications cover vulcanized compounds of natural rubber, reclaimed rubber, synthetic rubber, or rubber-like materials, alone or in combination, which are intended for use in manufacturing products for automotive and aeronautical applications. Compounds for tires, inner tubes, sponge rubber, and hard rubber are not included.

(b) The compounds listed in Tables I, II, III, IV, and V are of two types grouped in five classifications based primarily on physical properties. Although the individual compounds provide a wide variety and range of physical properties, it should be understood that

these specifications are general and are intended to furnish a basis for engineers to select material suitable for common applications. When using these classifications as a basis for detailed product specifications, care should be taken to choose those grades having only the properties required for the intended service. Since it is recognized that basic test requirements used mainly to classify compounds do not sufficiently describe some compositions required by the automotive and aeronautical industries, provision is made for added test requirements which are indicated by suitable suffixes to the grade numbers. When the compounds may be used for purposes where the requirements are too particular to be prescribed in general specifications, it is advisable for the purchaser to consult the manufacturer to secure adjustment of the properties to suit the actual operations to which the material is to be subjected.

(c) In case of conflict between the

<sup>1</sup> Under the standardization procedure of the A.S.T.M., these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by the A.S.T.M. at annual meeting, June, 1946.

provisions of these general specifications and those of detailed specifications or methods of test for a particular product, the latter shall take precedence.

### Types of Compounds

2. These specifications cover two types of compounds designated by the prefix letters R and S, as follows:

*Type R.*—Compounds made from natural rubber, reclaimed rubber, synthetic rubber, or rubber-like materials, alone or in combination, for services having no specific requirement for resistance to the action of petroleum-base oils or other organic fluids.

*Type S.*—Compounds made from synthetic rubber or rubber-like materials for services where resistance to the action of petroleum-base oils or other organic fluids is required.

### Classes and Grades of Compounds

3. (a) *Classes.*—Type R compounds are divided into two classes designated by the letters N and S added to the R prefix, and type S into three classes designated by the letters A, B, and C added to the S prefix, as follows:

*Type R: Class RN.*—Compounds containing natural rubber hydrocarbon either in the form of crude or reclaimed rubber to the extent of 50 per cent or more by volume of the total elastomer content. *No requirement for oil resistance.* *Class RS.*—Compounds containing synthetic rubber or rubber-like material to the extent of 50 per cent or more by volume of the total elastomer content. *No requirement for oil resistance.*

*Type S: Class SA.*—Compounds made from synthetic rubber or rubber-like material having *maximum* oil resistance. *Class SB.*—Compounds made from synthetic rubber or rubber-like material having *good* oil resistance. *Class SC.*—Compounds made from synthetic rubber

or rubber-like material having *medium* oil resistance.

(b) *Grades.*—Each class of compound has a number of different grades, each having different physical properties. The grades shall be designated by numbers following the prefix letters and, when necessary, by suffix letters after the grade number, as follows:

*Grade Numbers* shall consist of three digits, the first indicating the durometer hardness range, for example, 3 for  $30 \pm 5$ , 4 for  $40 \pm 5$ , etc. The next two digits indicate the minimum tensile strength, for example, 09 for 900 psi., 15 for 1500 psi., etc.

*Suffix Numbers* shall be used only in those cases where compounds having the same tensile strength may be specified with different ultimate elongations. They shall be single digits preceded by a hyphen and appended to the grade numbers indicating the required minimum elongations, for example, -4 for 400 per cent, -7 for 700 per cent, etc.

*Suffix Letters* may be added singly or in combination after any grade number to indicate additional requirements beyond those specified in Tables I to V as basic requirements for that particular grade. In those cases where a specific test for the property covered by a suffix letter has not been made a part of these specifications, the suffix letter is not to be used until suitable tests have been arranged by agreement between the manufacturer and the purchaser. The manufacturer will not be held accountable for special tests worked out by the purchaser unless the tests are specified on part drawings or the manufacturer has been notified in writing regarding these tests previous to entering into a contract to furnish these materials. The significance of the approved suffix letters is as follows:

## SIGNIFICANCE OF SUFFIX LETTERS

Suffix Letter	Suffix Letter
A. .... {Oven aging test required with values as specified in Tables I, II, IV, and V.	{Uniform adhesion values should range upward from approximately 200 psi. for compounds of less than 50 durometer hardness, and from approximately 250 psi. for compounds of over 50 durometer hardness, provided the compounds are designed for use in bonding to metal.
B. .... {Compression set test required with values as specified in Tables I, II, IV, and V.	{Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (A.S.T.M. Designation: D 429)* should be used when possible. However, it is usually desirable to run tests on the actual production parts, in which case, the adhesion value is measured by a tension pull normal to the plane of the metal surfaces and calculations are based on the projected area of the smaller metal part.
C. .... {Weather resistance required. Test to be arranged between the manufacturer and the purchaser.	{It is recommended that the respective purchaser and manufacturer of products requiring adhesion to metal mutually agree on the test method and adhesion value required for each specific application.
D. .... {Load deflection test required with values as specified in Tables I and II.	K. .... (continued)
E. .... {Oil aging test required with values as specified in Tables III, IV, and V.	
F. .... {Grades passing the low-temperature brittleness test (Section 8 (h)) at -40 F.	
FF. .... {Grades passing the low-temperature brittleness test (Section 8 (h)) at -70 F.	
G. .... {Resistance to tear required. Test to be arranged between the manufacturer and the purchaser.	L. .... {Resistance to water absorption required. Test to be arranged between the manufacturer and the purchaser.
H. .... {Resistance to severe flexing required. Test to be arranged between the manufacturer and the purchaser.	M. .... {Inflammability resistance required. Test to be arranged between the manufacturer and the purchaser.
J. .... {Resistance to abrasion required. Test to be arranged between the manufacturer and the purchaser.	N. .... {Resistance to impact required. Test to be arranged between the manufacturer and the purchaser.
K. .... {Adhesion to metal required. Natural and synthetic rubber compounds can be bonded to most metals. The bond obtained is dependent on many factors, such as: (1) Type of compound. (2) Type of metal. (3) Surface preparation of the metal. (4) Type of adhesive used. (5) Size and shape of the product. (6) Method of vulcanization.	P. .... {Test for the degree of staining of surface finishes required. Methods of test and limits to be arranged between the manufacturer and the purchaser.
	Z. .... {Special tests of either the usual laboratory type or performance type required. Such tests to be arranged between the manufacturer and the purchaser.

NOTE: *Example*.—Grade SB 625 F designates a synthetic compound with good oil resistance having a durometer hardness of  $60 \pm 5$ , and an original minimum tensile strength of 2500 psi., and which passes the low-temperature brittleness test at -40 F. and conforms to the other basic requirements prescribed in Table IV.

\* Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

TABLE I.—PHYSICAL REQUIREMENTS OF RUBBER COMPOUNDS TYPE R, CLASS RN, NON-OIL RESISTANT.

(See Section 8 for Methods of Testing.)

Basic Requirements					Requirements Added by Suffix Letters <sup>a</sup>			
Grade Number	Durometer Hardness Number	A.S.T.M. Hardness Number <sup>b</sup>	Tensile Strength, min., psi.	Ultimate Elongation, min., per cent	Suffix A		Suffix B	Suffix D
					Oven Aged 70 hr. at 158 F.			
					Change in Tensile Strength, max., per cent	Change in Ultimate Elongation, max., per cent	Compression Set, max., per cent	20 per cent Load Deflection psi.
†RN300.....	30 ± 5	150 ± 20	...	...	...	...	25	...
RN309.....	30 ± 5	150 ± 20	900	500	-25	-25	50	...
RN315.....	30 ± 5	150 ± 20	1 500	600	-25	-25	50	70 ± 10
RN320.....	30 ± 5	150 ± 20	2 000	600	-25	-25	50	70 ± 10
RN330.....	30 ± 5	150 ± 20	3 000	700	-25	-25	50	70 ± 10
*†RN400.....	40 ± 5	105 ± 15	...	...	...	...	25	...
RN405.....	40 ± 5	105 ± 15	500	300	-25	-25	50	...
*RN409.....	40 ± 5	105 ± 15	900	500	-25	-25	50	...
*RN415.....	40 ± 5	105 ± 15	1 500	500	-25	-25	50	100 ± 15
*RN420.....	40 ± 5	105 ± 15	2 000	500	-25	-25	50	100 ± 15
RN430.....	40 ± 5	105 ± 15	3 000	600	-25	-25	50	100 ± 15
*†RN500.....	50 ± 5	75 ± 10	...	...	...	...	25	...
*RN506.....	50 ± 5	75 ± 10	600	300	-25	-25	50	...
*RN509.....	50 ± 5	75 ± 10	900	300	-25	-25	50	...
*RN515.....	50 ± 5	75 ± 10	1 500	400	-25	-25	50	140 ± 20
*RN525.....	50 ± 5	75 ± 10	2 500	500	-25	-25	50	140 ± 20
RN535.....	50 ± 5	75 ± 10	3 500	600	-25	-25	50	140 ± 20
*†RN600.....	60 ± 5	55 ± 8	...	...	...	...	25	...
RN606.....	60 ± 5	55 ± 8	600	300	-25	-25	50	...
*RN609.....	60 ± 5	55 ± 8	900	300	-25	-25	50	...
*RN615.....	60 ± 5	55 ± 8	1 500	350	-25	-25	50	195 ± 30
RN620.....	60 ± 5	55 ± 8	2 000	400	-25	-25	50	195 ± 30
*RN625.....	60 ± 5	55 ± 8	2 500	450	-25	-25	50	195 ± 30
RN635.....	60 ± 5	55 ± 8	3 500	550	-25	-25	50	195 ± 30
†RN700.....	70 ± 5	40 ± 7	...	...	...	...	25	...
RN703.....	70 ± 5	40 ± 7	300	100	-25	-25	50	...
RN704.....	70 ± 5	40 ± 7	400	150	-25	-25	50	...
*RN706.....	70 ± 5	40 ± 7	600	200	-25	-25	50	...
RN709.....	70 ± 5	40 ± 7	900	250	-25	-25	50	...
*RN715.....	70 ± 5	40 ± 7	1 500	300	-25	-25	50	285 ± 50
*RN720.....	70 ± 5	40 ± 7	2 000	350	-25	-25	50	285 ± 50
RN730.....	70 ± 5	40 ± 7	3 000	450	-25	-25	50	285 ± 50
RN804.....	80 ± 5	28 ± 7	400	100	-25	-25	50	...
*RN806.....	80 ± 5	28 ± 7	600	150	-25	-25	50	...
RN809.....	80 ± 5	28 ± 7	900	200	-25	-25	50	...
*RN812.....	80 ± 5	28 ± 7	1 200	250	-25	-25	50	...
*RN820.....	80 ± 5	28 ± 7	2 000	250	-25	-25	50	450 ± 75
RN825.....	80 ± 5	28 ± 7	2 500	300	-25	-25	50	450 ± 75
*RN907.....	90 ± 5	13 ± 7	700	75	-25	-25	...	...
RN915.....	90 ± 5	13 ± 7	1 500	200	-25	-25	...	...

<sup>a</sup> See Section 3.<sup>b</sup> Required only for referee purposes.

\* The compounds marked with an asterisk are those which are most commonly used. It is suggested that these compounds be specified when possible.

† The compounds whose grade numbers are even hundreds are designed primarily for low compression set, and are for use in such applications as motor mounts, spring shackle bushings, rubber springs (vibration absorbers), brake cups, and many other applications where maximum resistance to permanent distortion under load is of paramount importance. Tensile and elongation requirements have been omitted because compression set is of first importance. Tensile and elongation cannot be specified in the tables because of the wide variation in these values.

TABLE II.—PHYSICAL REQUIREMENTS OF RUBBER COMPOUNDS, TYPE R, CLASS RS, NON-OIL RESISTANT.

(See Section 8 for Methods of Testing.)

Basic Requirements					Requirements Added by Suffix Letters <sup>a</sup>			
Grade Number	Durometer Hardness Number	A.S.T.M. Hardness Number <sup>b</sup>	Tensile Strength, min., psi.	Ultimate Elongation min., per cent	Suffix A		Suffix B	Suffix D
					Oven Aged 70 hr. at 158 F.			
					Change in Tensile Strength, max., per cent	Change in Ultimate Elongation, max., per cent	Compression Set, max., per cent	20 per cent Load Deflection, c psi.
†RS300.....	30 ± 5	175 ± 25	...	...	...	...	25	...
RS307.....	30 ± 5	175 ± 25	700	300	-25	-25	50	...
RS309.....	30 ± 5	175 ± 25	900	300	-25	-25	50	...
RS309-5.....	30 ± 5	175 ± 25	900	500	-25	-25	50	...
RS315.....	30 ± 5	175 ± 25	1 500	600	-25	-25	50	70 ± 15
RS320.....	30 ± 5	175 ± 25	2 000	500	-25	-25	50	70 ± 15
RS320-7.....	30 ± 5	175 ± 25	2 000	700	-25	-25	50	70 ± 15
†RS400.....	40 ± 5	125 ± 25	...	...	...	...	25	...
RS402.....	40 ± 5	125 ± 25	200	150	-25	-25	50	...
RS405.....	40 ± 5	125 ± 25	500	300	-25	-25	50	...
RS405-7.....	40 ± 5	125 ± 25	500	700	-25	-25	50	...
*RS409.....	40 ± 5	125 ± 25	900	400	-25	-25	50	...
RS415.....	40 ± 5	125 ± 25	1 500	500	-25	-25	50	102.5 ± 22.5
RS415-7.....	40 ± 5	125 ± 25	1 500	700	-25	-25	50	102.5 ± 22.5
RS420.....	40 ± 5	125 ± 25	2 000	700	-25	-25	50	102.5 ± 22.5
RS425.....	40 ± 5	125 ± 25	2 500	600	-25	-25	50	102.5 ± 22.5
*†RS500.....	50 ± 5	90 ± 20	...	...	...	...	25	...
RS503.....	50 ± 5	90 ± 20	300	125	-25	-25	50	...
RS506.....	50 ± 5	90 ± 20	600	300	-25	-25	50	...
*RS509.....	50 ± 5	90 ± 20	900	300	-25	-25	50	...
RS509-5.....	50 ± 5	90 ± 20	900	500	-25	-25	50	...
*RS515.....	50 ± 5	90 ± 20	1 500	400	-25	-25	50	145 ± 30
RS515-6.....	50 ± 5	90 ± 20	1 500	600	-25	-25	50	145 ± 30
RS518.....	50 ± 5	90 ± 20	1 800	400	-25	-25	50	145 ± 30
RS520.....	50 ± 5	90 ± 20	2 000	600	-25	-25	50	145 ± 30
*†RS600.....	60 ± 5	63 ± 17	...	...	...	...	25	...
RS604.....	60 ± 5	63 ± 17	400	100	-25	-25	50	...
RS606.....	60 ± 5	63 ± 17	600	300	-25	-25	50	...
*RS609.....	60 ± 5	63 ± 17	900	300	-25	-25	50	...
RS612.....	60 ± 5	63 ± 17	1 200	500	-25	-25	50	...
*RS615.....	60 ± 5	63 ± 17	1 500	350	-25	-25	50	205 ± 45
*RS620.....	60 ± 5	63 ± 17	2 000	600	-25	-25	50	205 ± 45
RS625.....	60 ± 5	63 ± 17	2 500	450	-25	-25	50	205 ± 45
†RS700.....	70 ± 5	42 ± 12	...	...	...	...	25	...
RS703.....	70 ± 5	42 ± 12	300	100	-25	-25	50	...
RS704.....	70 ± 5	42 ± 12	400	150	-25	-25	50	...
RS706.....	70 ± 5	42 ± 12	600	200	-25	-25	50	...
*RS709.....	70 ± 5	42 ± 12	900	250	-25	-25	50	...
RS709-4.....	70 ± 5	42 ± 12	900	400	-25	-25	50	...
*RS715.....	70 ± 5	42 ± 12	1 500	300	-25	-25	50	302.5 ± 72.5
RS715-5.....	70 ± 5	42 ± 12	1 500	500	-25	-25	50	302.5 ± 72.5
RS720.....	70 ± 5	42 ± 12	2 000	350	-25	-25	50	302.5 ± 72.5
RS725.....	70 ± 5	42 ± 12	2 500	300	-25	-25	50	302.5 ± 72.5
RS804.....	80 ± 5	30 ± 10	400	50	-25	-25	50	...
*RS806.....	80 ± 5	30 ± 10	600	150	-25	-25	50	...
RS809.....	80 ± 5	30 ± 10	900	200	-25	-25	50	...
*RS812.....	80 ± 5	30 ± 10	1 200	250	-25	-25	50	...
RS820.....	80 ± 5	30 ± 10	2 000	200	-25	-25	50	472.5 ± 102.5
RS825.....	80 ± 5	30 ± 10	2 500	300	-25	-25	50	472.5 ± 102.5
RS907.....	90 ± 5	15 ± 10	700	75	-25	-25	...	...
*RS915.....	90 ± 5	15 ± 10	1 500	75	-25	-25	...	...

<sup>a</sup> See Section 3.<sup>b</sup> Required only for referee purposes.

<sup>c</sup> Load deflection values are not provided for compounds having a tensile strength of less than 1500 psi. nor for compounds over 80 durometer hardness.

\* The compounds marked with an asterisk are those which are most commonly used. It is suggested that these compounds be specified when possible.

† The compounds whose grade numbers are even hundreds are designed primarily for low compression set, and are for use in such applications as motor mounts, spring shackle bushings, rubber springs (vibration absorbers), brake cups, and many other applications where maximum resistance to permanent distortion under load is of paramount importance. Tensile and elongation requirements have been omitted because compression set is of first importance. Tensile and elongation cannot be specified in the tables because of the wide variation in these values.



## Composition and Manufacture

4. Compounds furnished under these specifications shall be manufactured from natural rubber, reclaimed rubber, synthetic rubber, or rubber-like materials together with added compounding ingredients of such nature and quantity as not to prevent compliance with the specified requirements. In permitting choice in use of those materials by the manufacturer, it is not intended to imply that the different rubber materials are equivalent with respect to all

free from porous areas, weak sections, bubbles, foreign matter or other defects affecting serviceability.

## Color

5. Unless otherwise specified, the rubber compounds shall be black and free from objectionable bloom.

## Physical Properties

6. The various grades of compounds shall conform to the requirements as to physical properties prescribed in Tables I, II, III, IV, and V, together with any

TABLE III.—PHYSICAL REQUIREMENTS OF SYNTHETIC RUBBER COMPOUNDS, TYPE S, CLASS SA, OIL RESISTANT.

(See Section 8 for Methods of Testing.)

Basic Requirements						Requirements Added by Suffix Letters <sup>a</sup>			
Grade Number	Durometer Hardness Number	A.S.T.M. Hardness Number <sup>b</sup>	Tensile Strength, min., psi.	Ultimate Elongation, min., per cent	Oil Aged 70 hr. at 158 F., Change in Volume (Limits), per cent	Suffix E		Suffix A	Suffix B
						Oil Aged 70 hr. at 158 F.		Oven Aged 70 hr. at 158 F.	Compression Set
						Change in Tensile Strength, max., per cent	Change in Durometer Hardness (Limits)		
SA302.....	30 ± 5	150 ± 20	200	500	-15 to +5	-20	-5 to +10	Values for oven aging and compression set tests are omitted for class SA compounds as these materials may be thermoplastic.	
SA403.....	40 ± 5	105 ± 15	300	500	-5 to +3	-20	-5 to +5		
SA506.....	50 ± 5	75 ± 10	600	500	-3 to +3	-20	-5 to 0		
SA607.....	60 ± 5	55 ± 8	700	400	-3 to +3	-20	-5 to 0		
SA706.....	70 ± 5	40 ± 7	600	400	-3 to +3	-20	-5 to 0		
SA709.....	70 ± 5	40 ± 7	900	300	-3 to +3	-20	-5 to 0		
SA809.....	80 ± 5	28 ± 7	900	200	-3 to +3	-20	-5 to 0		
SA910.....	90 ± 5	13 ± 7	1000	100	-3 to +3	-20	-5 to 0		

<sup>a</sup> See Section 3.

<sup>b</sup> Required only for referee purposes.

physical properties. Any special characteristics other than those prescribed in these specifications, which may be desired for specific applications (for example, resistance to ozone, flexing, abrasion, or specific fluids) shall be specified in the product specifications, as they may influence the choice of the type of rubber material or other ingredient used. The compounds shall be vulcanized by a suitable heat treatment. All materials and workmanship shall be in accordance with good commercial practice and the resulting stock shall be

additional requirements indicated by suffix letters in the grade designations as described in Section 3.

## Sampling

7. (a) The values for the physical requirements prescribed in these specifications define the properties of the compounds after vulcanization in molds in the form of standard laboratory test slabs or blocks prepared in accordance with the Standard Methods of Sample Preparation for Physical Testing of

Rubber Products (A.S.T.M. Designation: D 15).<sup>3</sup> Frequently the same compound will have differing properties when processed in different manners as required in various products. Therefore,

(b) When proof of conformance with these specifications is required for any lot of products in which their use was specified, the manufacturer shall, upon request of the purchaser at the time of

TABLE IV.—PHYSICAL REQUIREMENTS OF SYNTHETIC RUBBER COMPOUNDS, TYPE S, CLASS SB, OIL RESISTANT.

(See Section 8 for Methods of Testing.)

Basic Requirements						Requirements Added by Suffix Letters <sup>a</sup>							
Grade Number	Durometer Hardness Number	A.S.T.M. Hardness Number <sup>b</sup>	Tensile Strength, min., psi.	Ultimate Elongation, min., per cent	Oil Aged 70 hr. at 212 F., Change in Volume (Limits), per cent	Suffix E		Suffix A				Suffix B	
						Oil Aged 70 hr. at 212 F.		Oven Aged 70 hr. at 212 F.				Compression Set, max., per cent	
						Change in Tensile Strength, max., per cent	Change in Durometer Hardness (Limits)	Change in Tensile Strength, max., per cent	Change in Ultimate Elongation, max., per cent	Change in Durometer Hardness, (Limits)		22 hr. at 158 F.	70 hr. at 212 F.
SB305..	30 ± 5	150 ± 20	500	400	-15 to +25	-20	-10 to +20	-20	-50	0 to +20		50	...
SB309..	30 ± 5	150 ± 20	900	500	-15 to +25	-20	-10 to +20	-20	-50	0 to +20		50	...
†SB400..	40 ± 5	105 ± 15	...	...	-15 to +25	...	-10 to +15	...	...	0 to +15		...	70
SB405..	40 ± 5	105 ± 15	500	400	-15 to +25	-20	-10 to +20	-20	-50	0 to +20		45	...
*SB410..	40 ± 5	105 ± 15	1000	500	-15 to +25	-20	-10 to +20	-20	-50	0 to +20		45	...
*SB415..	40 ± 5	105 ± 15	1500	500	-15 to +25	-20	-10 to +15	-20	-50	0 to +15		45	...
*†SB500..	50 ± 5	75 ± 10	...	...	-5 to +25	...	-10 to +10	...	...	0 to +10		...	65
SB508..	50 ± 5	75 ± 10	800	400	-5 to +25	-20	-10 to +15	-20	-50	0 to +15		40	...
*SB515..	50 ± 5	75 ± 10	1500	500	-5 to +25	-20	-10 to +15	-20	-50	0 to +15		40	...
SB520..	50 ± 5	75 ± 10	2000	500	-5 to +25	-20	-10 to +10	-20	-50	0 to +10		40	...
*†SB600..	60 ± 5	55 ± 8	...	...	-5 to +25	...	-10 to +10	...	...	0 to +10		...	65
SB608..	60 ± 5	55 ± 8	800	300	-5 to +25	-20	-10 to +10	-20	-60	0 to +15		35	...
*SB612..	60 ± 5	55 ± 8	1200	300	-5 to +25	-20	-10 to +10	-20	-60	0 to +15		35	...
*SB617..	60 ± 5	55 ± 8	1700	400	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		35	...
SB625..	60 ± 5	55 ± 8	2500	500	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		35	...
*†SB700..	70 ± 5	40 ± 7	...	...	-5 to +25	...	-10 to +10	...	...	0 to +10		...	65
SB710..	70 ± 5	40 ± 7	1000	300	-5 to +25	-20	-10 to +10	-20	-80	0 to +10		30	...
*SB715..	70 ± 5	40 ± 7	1500	300	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
SB720..	70 ± 5	40 ± 7	2000	300	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
SB725..	70 ± 5	40 ± 7	2500	300	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
SB730..	70 ± 5	40 ± 7	3000	400	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
†SB800..	80 ± 5	28 ± 7	...	...	-5 to +25	...	-10 to +5	...	...	0 to +10		...	65
SB804..	80 ± 5	28 ± 7	400	100	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
SB810..	80 ± 5	28 ± 7	1000	150	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
*SB815..	80 ± 5	28 ± 7	1500	150	-5 to +25	-20	-10 to +10	-20	-60	0 to +10		30	...
SB820..	80 ± 5	28 ± 7	2000	150	-5 to +25	-20	-10 to +5	-20	-60	0 to +10		30	...
SB902..	90 ± 5	13 ± 7	200	50	-5 to +25	-20	-10 to +5	-20	-60	0 to +5		40	...
*SB910..	90 ± 5	13 ± 7	1000	100	-5 to +25	-20	-10 to +5	-20	-60	0 to +5		30	...

<sup>a</sup> See Section 3.

<sup>b</sup> Required only for referee purposes.

\* The compounds marked with an asterisk are those which are most commonly used. It is suggested that these compounds be specified when possible.

† The compounds whose grade numbers are even hundreds are designed primarily for low compression set, and are for use in such applications as motor mounts, spring shackle bushings, rubber springs (vibration absorbers), brake cups, and many other applications where maximum resistance to permanent distortion under load is of paramount importance. Tensile and elongation requirements have been omitted because compression set is of first importance. Tensile and elongation cannot be specified in the tables because of the wide variation in these values.

samples taken from finished products cannot be expected in all cases to meet the values prescribed.

ordering, furnish a sufficient number of test slabs, having dimensions 6 by 6 by 0.080 ± 0.010 in. and test blocks 2 by 4

by  $0.55 \pm 0.02$  in. for the proper performance of the required tests. These samples shall be guaranteed to have equivalent cure and to be from the same

### Methods of Testing

8. The properties enumerated in these specifications shall be determined in

TABLE V.—PHYSICAL REQUIREMENTS OF SYNTHETIC RUBBER COMPOUNDS, TYPE S, CLASS SC, OIL RESISTANT.

(See Section 8 for Methods of Testing.)

Basic Requirements						Requirements Added by Suffix Letters <sup>a</sup>						
Grade Number	Durometer Hardness Number	A.S.T.M. Hardness Number <sup>b</sup>	Tensile Strength, min., psi.	Ultimate Elongation, min., per cent	Oil Aged 70 hr. at 212 F., Change in Volume (Limits), per cent	Suffix E		Suffix A			Suffix B	
						Oil Aged 70 hr. at 212 F.		Oven Aged 70 hr. at 212 F.			Compression Set, max., per cent	
						Change in Tensile Strength, max., per cent	Change in Durometer Hardness (Limits)	Change in Tensile Strength, max., per cent	Change in Ultimate Elongation, max., per cent	Change in Durometer Hardness, (Limits)	22 hr. at 158 F.	70 hr. at 212 F.
SC305..	30 $\pm$ 5	150 $\pm$ 20	500	500	100 to 150	-85	-30 to -10	-15	-40	0 to +15	70	...
SC310..	30 $\pm$ 5	150 $\pm$ 20	1000	500	100 to 150	-85	-30 to -10	-15	-40	0 to +15	70	...
SC315..	30 $\pm$ 5	150 $\pm$ 20	1500	500	100 to 150	-85	-30 to -10	-15	-40	0 to +15	70	...
SC320..	30 $\pm$ 5	150 $\pm$ 20	2000	500	100 to 150	-85	-30 to -10	-15	-40	0 to +15	70	...
†SC400..	40 $\pm$ 5	105 $\pm$ 15	...	...	90 to 140	...	-35 to -15	...	...	0 to +15	...	75
*SC405..	40 $\pm$ 5	105 $\pm$ 15	500	500	90 to 140	-80	-35 to -15	-15	-40	0 to +15	65	...
*SC415..	40 $\pm$ 5	105 $\pm$ 15	1500	500	90 to 140	-80	-35 to -15	-15	-40	0 to +15	65	...
*SC420..	40 $\pm$ 5	105 $\pm$ 15	2000	500	90 to 140	-80	-35 to -15	-15	-40	0 to +15	60	...
SC425..	40 $\pm$ 5	105 $\pm$ 15	2500	500	90 to 140	-80	-35 to -15	-15	-40	0 to +15	60	...
*†SC500..	50 $\pm$ 5	75 $\pm$ 10	...	...	80 to 130	...	-40 to -20	...	...	0 to +15	...	70
SC507..	50 $\pm$ 5	75 $\pm$ 10	700	300	80 to 130	-70	-40 to -20	-15	-40	0 to +15	60	...
*SC512..	50 $\pm$ 5	75 $\pm$ 10	1200	300	80 to 130	-70	-40 to -20	-15	-40	0 to +15	60	...
*SC520..	50 $\pm$ 5	75 $\pm$ 10	2000	400	80 to 130	-70	-40 to -20	-15	-40	0 to +15	50	...
SC525..	50 $\pm$ 5	75 $\pm$ 10	2500	400	80 to 130	-70	-40 to -20	-15	-40	0 to +15	50	...
SC530..	50 $\pm$ 5	75 $\pm$ 10	3000	500	80 to 130	-70	-40 to -20	-15	-40	0 to +15	50	...
SC600..	60 $\pm$ 5	55 $\pm$ 8	...	...	70 to 120	...	-50 to -20	...	...	0 to +10	...	70
*SC608..	60 $\pm$ 5	55 $\pm$ 8	800	300	70 to 120	-60	-50 to -20	-15	-40	0 to +15	55	...
*SC614..	60 $\pm$ 5	55 $\pm$ 8	1400	300	70 to 120	-60	-50 to -20	-15	-40	0 to +10	55	...
*SC620..	60 $\pm$ 5	55 $\pm$ 8	2000	300	70 to 120	-60	-50 to -20	-15	-40	0 to +10	50	...
SC625..	60 $\pm$ 5	55 $\pm$ 8	2500	300	70 to 120	-60	-50 to -20	-15	-40	0 to +10	50	...
SC630..	60 $\pm$ 5	55 $\pm$ 8	3000	400	70 to 120	-60	-50 to -20	-15	-40	0 to +10	45	...
*†SC700..	70 $\pm$ 5	40 $\pm$ 7	...	...	60 to 110	...	-50 to -25	...	...	0 to +10	...	65
SC707..	70 $\pm$ 5	40 $\pm$ 7	700	200	60 to 110	-60	-50 to -25	-15	-40	0 to +10	50	...
*SC717..	70 $\pm$ 5	40 $\pm$ 7	1700	200	60 to 110	-60	-50 to -25	-15	-40	0 to +10	45	...
SC725..	70 $\pm$ 5	40 $\pm$ 7	2500	300	60 to 110	-60	-50 to -25	-15	-40	0 to +10	40	...
†SC800..	80 $\pm$ 5	28 $\pm$ 7	...	...	50 to 100	-40	-50 to -30	...	...	0 to +10	...	65
SC804..	80 $\pm$ 5	28 $\pm$ 7	400	100	50 to 100	-40	-50 to -30	-15	-40	0 to +10	50	...
SC808..	80 $\pm$ 5	28 $\pm$ 7	800	100	50 to 100	-40	-50 to -30	-15	-40	0 to +10	50	...
*SC815..	80 $\pm$ 5	28 $\pm$ 7	1500	100	50 to 100	-40	-50 to -30	-15	-40	0 to +10	45	...
SC902..	90 $\pm$ 5	13 $\pm$ 7	200	50	40 to 90	-30	-50 to -30	-15	-40	0 to +5	50	...
*SC910..	90 $\pm$ 5	13 $\pm$ 7	1000	100	40 to 90	-30	-50 to -30	-15	-40	0 to +5	50	...

<sup>a</sup> See Section 3.

<sup>b</sup> Required only for referee purposes.

\* The compounds marked with an asterisk are those which are most commonly used. It is suggested that these compounds be specified when possible.

† The compounds whose grade numbers are even hundreds are designed primarily for low compression set, and are for use in such applications as motor mounts, spring shackle bushings, rubber springs (vibration absorbers), brake cups, and many other applications where maximum resistance to permanent distortion under load is of paramount importance. Tensile and elongation requirements have been omitted because compression set is of first importance. Tensile and elongation cannot be specified in the tables because of the wide variation in these values.

run or batch of compound used in the lot. Unless otherwise specified, a lot shall consist of all products of the same composition and the same grade submitted for inspection at the same time.

accordance with the following methods of the American Society for Testing Materials except as modified in accordance with certain of the references given below. All exposure periods and tem-

peratures prescribed in Tables I, II, III, IV, and V shall be given precedence over those specified in the A.S.T.M. methods.

NOTE.—Before any tests are started, the classifications of rubber compounds, and the A.S.T.M. methods of testing referred to should be studied carefully.

(a) *A.S.T.M. Hardness*.—Standard Method of Test for Hardness of Rubber (A.S.T.M. Designation: D 314).<sup>3</sup>

(b) *Durometer Hardness*.—Tentative Method of Test for Indentation of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

(c) *Tensile Strength and Elongation*.—Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412),<sup>3</sup> except that the test specimens shall be 0.070 to 0.090 in. in thickness and shall be cut using die C.

(d) *Load Deflection*.—Standard Methods of Test for Compression-Deflection Characteristics of Vulcanized Rubber (A.S.T.M. Designation: D 575).<sup>3</sup>

(e) *Compression Set*.—Method B of the Tentative Methods of Test for Compression Set of Vulcanized Rubber (A.S.T.M. Designation: D 395)<sup>3</sup> in which the compression is at constant deflection and the percentage of set is based on the original deflection of the specimen rather than the original thickness. The percentage of compression employed shall depend upon the hardness of the rubber being tested, according to the following requirements:

Durometer Hardness Number	Deflection, per cent of original thickness
30 $\pm$ 5	40
40 $\pm$ 5	40
50 $\pm$ 5	30
60 $\pm$ 5	30
70 $\pm$ 5	25
80 $\pm$ 5	25
90 $\pm$ 5	20

(f) *Oven Aging*.—Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573).<sup>3</sup> It is

essential that the oven used shall have means of forced-air circulation and adequate temperature control as required by Section 4 (h) of A.S.T.M. Methods D 573. In addition to determining the percentage changes in tensile strength and elongation, the following determination shall be made on type S compounds:

*Durometer Hardness*.—After the specimen has been aged and cooled, the durometer hardness number shall be determined in accordance with A.S.T.M. Method D 676 and the change from the original hardness number noted. The specimen shall have the minimum thickness specified in A.S.T.M. Method D 676, built up if necessary from thinner sections. The specimen shall be large enough to allow at least  $\frac{1}{2}$  in. in any direction from the indentation point to the edge of the specimen.

(g) *Liquid Aging*.—Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (A.S.T.M. Designation: D 471),<sup>3</sup> using the procedure described in Section 10 (c) of Methods D 471 with the following modifications:

(1) *Test Liquid*.—Petroleum-base oil<sup>4</sup> No. 3 specified in A.S.T.M. Methods D 471 shall be used.

(2) *Condenser*.—An air condenser shall be used in all cases. This condenser shall be a glass tube 30 in. in length with a  $\frac{1}{2}$ -in. bored fitted tightly to the immersion tube by means of a cork stopper. The bottom of the condenser tube shall project  $\frac{1}{2}$  in. below the stopper.

(3) *Volume of Liquid*.—The test tube prescribed in A.S.T.M. Methods D 471 shall be three quarters full of liquid after the test specimens are immersed.

<sup>4</sup>The oil known as "Circo light," or equivalent, is satisfactory.

(4) *Tensile Strength*.—The tensile strength of the specimen after removal from the liquid, shall be calculated on the original unaged cross-sectional area, and not according to the formula given in Section 11 (c) of A.S.T.M. Methods D 471.

(5) *Durometer Hardness*.—Immediately after removal of the specimen from the cool liquid, and after drying as specified, the durometer hardness number shall be determined in accordance with A.S.T.M. Method D 676, and the change from the original durometer hardness number noted.

(h) *Low-Temperature Brittleness*.—Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736).<sup>3</sup> Type R compounds shall be given an exposure period of 96 hr. and type S compounds of classes SA, SB, and SC shall have an exposure period of 5 hr. All grades carrying the suffix letter F in the grade number shall be exposed at -40 F., and those with suffix letters FF at -70 F.

## Inspection and Rejection

9. (a) All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. The manufacturer shall afford the inspector all reasonable facilities, without charge, for tests and inspection.

(b) The purchaser may make the tests and inspection to govern acceptance or rejection of the material at his laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser and not later than 60 days after receipt of the material.

(c) All samples for testing, provided as specified in Section 7, shall be visually inspected to determine compliance with the material, workmanship, color, and nonblooming requirements.

(d) Any material which fails to conform to one or more of the test requirements may be retested at the expense of the manufacturer. For this purpose two additional tests shall be made for the requirement in which failure occurred. Failure of either of the retests shall be cause for final rejection.

(e) Rejected material shall be disposed of as directed by the manufacturer and at his expense.

# *Tentative Specifications for*

## NATURAL RUBBER CUPS FOR USE IN HYDRAULIC ACTUATING CYLINDERS<sup>1</sup>



**A.S.T.M. Designation: D 818 - 45 T**

ISSUED, 1945.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Headquarters of the A.S.T.M., 1916 Race St., Philadelphia 3, Pa., or the S.A.E., 29 W. 39th. St., New York 18, N.Y.

*These specifications were prepared jointly by the Society of Automotive Engineers and the American Society for Testing Materials.*

### Scope

1. These specifications cover molded cups compounded from natural rubber for use in hydraulic actuating cylinders employing hydraulic brake fluid of non-mineral oil type.

NOTE.—These specifications do not include requirements relating to chemical composition, tensile strength and elongation of the rubber compounds and materials composing the cup. The preparation of suitable specimens for tensile strength and elongation tests from a cup herein specified is impracticable; furthermore, separate test slabs prepared from rubber compounds representing the cups are not considered suitable.

### Resistance to Fluids at Elevated Temperature

2. After being subjected to the test for resistance to fluids at elevated temperatures as prescribed in Sections 13 to 16, the cups shall conform to the following requirements as to changes in dimensions and durometer hardness:

Change in volume.....	0 to +20 per cent
Change in outside diameter {	Lip..... 0 to +5.75 per cent (0 to +0.075 in.)
Base.....	0 to +5.75 per cent (0 to +0.075 in.)
Change in durometer hardness	-10 to +5 points

### Effect of Fluid Pressure at Elevated Temperature (Heat Pressure Test)

3. After being subjected to the heat pressure test prescribed in Sections 17 to 20 the cups shall conform to the following requirements as to changes in dimensions and durometer hardness:

#### MEASUREMENTS AFTER 30 MIN.

Change in outside diameter {	Lip..... -2 to +2 per cent (-0.025 to +0.025 in.)
Base.....	+3 per cent max. (+0.040 in. max.)
Change in durometer hardness	-5 to +0 points

#### MEASUREMENTS AFTER 48 HR.

Change in outside diameter {	Lip..... -3 per cent to +2 per cent (-0.040 to +0.025 in.)
Base.....	+3 per cent max. (+0.040 in. max.)
Change in durometer hardness	-5 to +0 points

### Stroking Test

4. (a) There shall be no leakage of fluid past the primary or secondary cups during the stroking test.

(b) After the stroking test, specimens shall be examined for wear and general condition which may affect their life and performance.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, June, 27, 1945.

(c) The change in durometer hardness after the stroking test shall be within the limits of minus 7 to plus 5 points from the original reading.

#### Low-Temperature Test

5. (a) After being held for 120 hr. at  $-40$  to  $-45$  F., the durometer hardness of the specimen shall not increase more than 25 points over the reading taken at room temperature.

(b) After the bend test, made while the specimen is held at a temperature of  $-40$  to  $-45$  F. following an exposure at this temperature for 120 hr., the cup shall return to its approximate original shape within 1 min.

(c) No leakage of fluid shall occur past the primary, secondary or wheel-cylinder cups during the low-temperature leakage test.

#### Aging

6. (a) The change in durometer hardness shall be within the limits of 0 to plus 5 points over the reading taken before aging.

(b) After the aging test, the specimens shall be examined for general condition and defects which may affect their life and performance.

#### Workmanship and Finish

7. Cups shall be free of blisters, pin holes, cracks, protuberances, embedded foreign matter or other physical defects which can be detected by thorough inspection and shall conform to the dimensions specified on the drawings. The sealing lip shall be sharp and free from any irregularities.

#### Marking

8. The identification mark of the manufacturer, cup size, and other details as specified on the drawings shall be molded into each cup.

#### Packaging

9. Cups shall be packaged to meet the requirements specified by the purchaser.

#### Inspection and Rejection

10. (a) All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. The manufacturer shall afford the inspector all reasonable facilities, without charge, for tests and inspection.

(b) The purchaser may make the tests and inspection to govern the acceptance or rejection of the cups at his laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser and not later than 60 days after receipt of the material.

(c) Any cups which fail to conform to one or more of the test requirements may be retested at the expense of the manufacturer. For this purpose two additional tests shall be made for the requirement in which failure occurred. Failure of either of the retests shall be cause for final rejection.

(d) All rejected cups shall be destroyed by the manufacturer, by cutting each cup or grinding through a rubber mill to insure that no substandard cups will be used.

#### METHODS OF TESTING

##### Sampling

11. (a) A quantity of 5000 cups of one size shall represent the minimum lot on which complete tests shall be conducted. The frequency and type of tests used to control production shall be agreed upon by the manufacturer and the purchaser.

(b) Specimens for resistance to fluid and heat pressure tests shall be wheel cylinder cups  $1\frac{1}{4}$  in. in diameter, and for stroking tests shall be primary and secondary cups  $1\frac{1}{8}$  in. in diameter. When conditions do not permit the use of these sizes, tests shall be made on cups of available size, in which case the per-



centage limits of the specifications shall apply.

(c) Cups used for purpose of tests shall not be more than six months old from the date of manufacture.

### Test Fluids

12. The fluid used in the tests shall be one of the standard non-mineral oil brake fluids as follows: Chrysler "ISO Brake," Delco No. 9, Ford M-3833-A, Wagner No. 21, and fluids approved in Army Ordinance Specification 2-111, latest issue.

### *Resistance to Fluids at Elevated Temperature*

### Apparatus

13. (a) *Micrometer*.—Micrometer calipers or other suitable apparatus to measure accurately in thousandths of an inch the lip and base diameters of the cup.

(b) *Containers*.—Tightly sealed containers of  $\frac{1}{2}$ -pt. capacity.

(c) *Oven*.—Any well-designed uniformly heated standard dry-air oven conforming to the requirements prescribed in Section 4 of the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573),<sup>3</sup> may be used. The oven shall be provided with proper temperature controls to maintain a standard test temperature of  $158 \pm 2$  F. Satisfactory circulation of air shall be secured by means of a fan.

### Test Specimens

14. Two cups shall be selected for test specimens (see Section 11 (b)).

### Procedure

15. (a) The specimens shall be wiped with an alcohol-saturated lint-free cloth to remove dirt and packing debris.

(b) The volume of each specimen shall be determined in accordance with Section

9 (a) of the Tentative Methods of Test for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (A.S.T.M. Designation: D 471),<sup>3</sup> except that a Jolly balance may be used.

(c) The diameters of the lip and base of the specimen shall be measured to the nearest thousandth of an inch, taking the average of two readings at right angles. Care shall be taken when measuring the base diameter that the measurement is taken within 0.015 in. from the back and parallel to the base of the specimen.

(d) The durometer hardness of the specimen shall be determined in accordance with Section 30. Two specimens shall be put in a  $\frac{1}{2}$ -pt. container containing 75 ml. of the specified test fluid and the container shall be sealed to prevent loss of vapor during the test. The container shall be placed in the oven at  $158 \pm 2$  F. for 120 hr. At the end of the heating period the container shall be taken from the oven and the test specimens removed immediately and wiped dry with an alcohol-saturated lint-free cloth.

(e) The test specimens shall be cooled for 30 min. on a wooden table top. The final volume, dimensions, and durometer hardness of each specimen shall then be determined as described in Paragraphs (b) to (d).

### Calculations and Report

16. (a) *Volume Change*.—The change in volume shall be calculated as follows, and reported:

Increase in volume, per cent =

$$\frac{(W_3 - W_4) - (W_1 - W_2)}{W_1 - W_2} \times 100$$

where:

$W_1$  = initial weight in air,

$W_2$  = initial weight in water,

$W_3$  = weight in air after immersion in the test fluid, and

$W_4$  = weight in water after immersion in the test fluid.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) *Dimensional Change*.—The original measurements shall be subtracted from the measurements taken after the test and the difference reported in thousandths of an inch and as a percentage. Any observable changes in the condition of the specimens shall also be reported.

(c) *Durometer Hardness*.—The difference between the original durometer hardness reading and the reading after the test shall be reported.

of the oven by suitable supports. A suitable oven is described in Section 4 of A.S.T.M. Method D 573.

(b) *Heat Pressure Device*.—Suitable means shall be provided to maintain a pressure of 500 psi. in cylinders used in the heat pressure test. The apparatus shown in Figs. 1 and 2 has been found satisfactory.

#### Test Specimens

18. Two wheel cylinder cups shall be

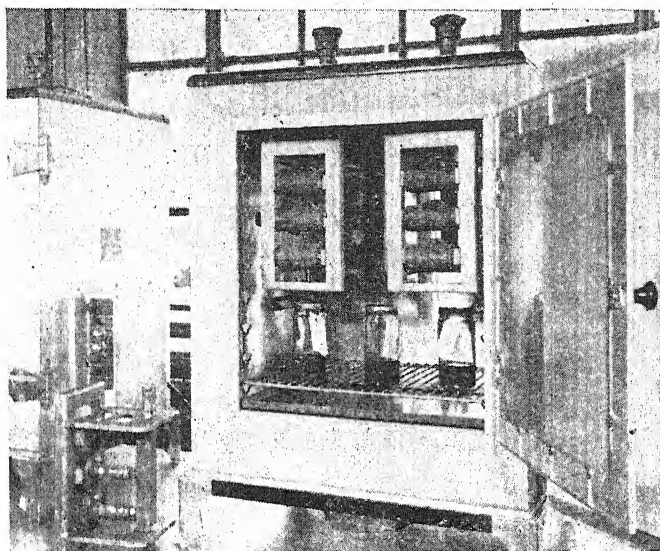


FIG. 1.—Oven Showing Fixtures for Heat Pressure Test, and Containers for Fluid Resistance Test.

#### Heat Pressure Test

##### Apparatus

17. (a) *Oven*.—Any well-designed uniformly heated standard dry-air oven of sufficient size to hold the heat pressure device may be used. The oven shall be provided with proper temperature controls to maintain a standard test temperature of  $158 \pm 2$  F. Satisfactory circulation of air shall be secured by means of a fan. The heat pressure device shall be centrally located in the oven and supported above the bottom

selected for test specimens (see Section 11 (b)).

##### Procedure

19. (a) The specimens shall be wiped with an alcohol-saturated lint-free cloth to remove dirt and packing debris.

(b) The diameters of the lip and base of the specimen shall be measured to the nearest thousandth of an inch, taking the average of two readings at right angles. Care shall be taken when measuring the base diameter that the measurement is taken within 0.015 in. from the

back and parallel to the base of the specimen.

(c) The durometer hardness of the specimens shall be determined in accordance with Section 30.

(d) The specimens shall be installed in a wheel cylinder, and connected to a master cylinder with weighted arm (see Figs. 1 and 2 for typical arrangement of

and wiped clean with an alcohol-saturated lint-free cloth.

(e) The specimens shall be cooled for 30 min. on a wooden table top, after which measurements of the dimensions and durometer hardness shall be taken as prescribed in Paragraphs (b) and (c).

(f) The specimens shall then be rested for 48 hr. on a wooden table top at room temperature after which the measurements of the dimensions and durometer hardness shall be repeated.

### Calculations and Report

20. The changes in dimensions and durometer hardness shall be calculated and reported as described in Section 16 (b) and (c).

### Stroking Test

#### Apparatus

21. (a) *Stroking Machine*.—The stroking machine shall consist of a suitable mechanical means for actuating the master cylinder containing the test specimens, which shall be operated at  $1000 \pm 100$  strokes per hour with a total piston movement of  $1\frac{7}{8}$  in. The initial movement of  $\frac{5}{8}$  in. to  $1\frac{1}{8}$  in. shall be at a pressure of 40 to 50 psi. to permit the primary cup to pass over the compensating port hole, and the balance of the stroke shall be at a test pressure of  $500 \pm 50$  psi., held constant by an adjustable relief valve. If master cylinder sizes other than those recommended are used, there shall be a minimum total movement of the master-cylinder piston of  $1\frac{1}{8}$  in. with a minimum stroke of  $\frac{1}{2}$  in. after the primary cup passes over the compensating port hole. The master cylinder shall be located in a uniformly heated dry-air oven maintained at the standard test temperature of  $158 \pm 2$  F. The equipment shown in Figs. 3 and 4 has been found satisfactory.

(b) *Alternative Stroking Machine*.—One hydraulic brake system master

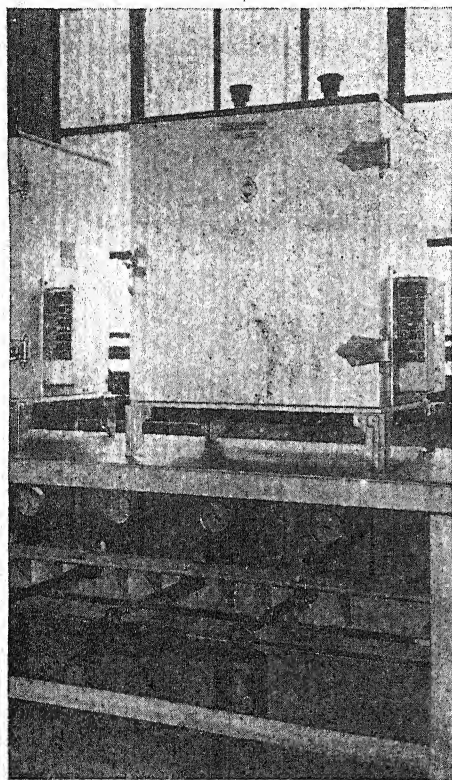


FIG. 2.—Oven Showing Weighted Master Cylinder for Applying Pressure in Heat Pressure Test.

equipment). The system shall be filled with the specified test fluid and all air expelled by bleeding. The weight on the master cylinder arm shall be adjusted to maintain a pressure of  $500 \pm 50$  psi. in the system. The test shall be continued for 120 hr. at  $158 \pm 2$  F. At the termination of the heating period, the specimens shall be removed from the cylinder

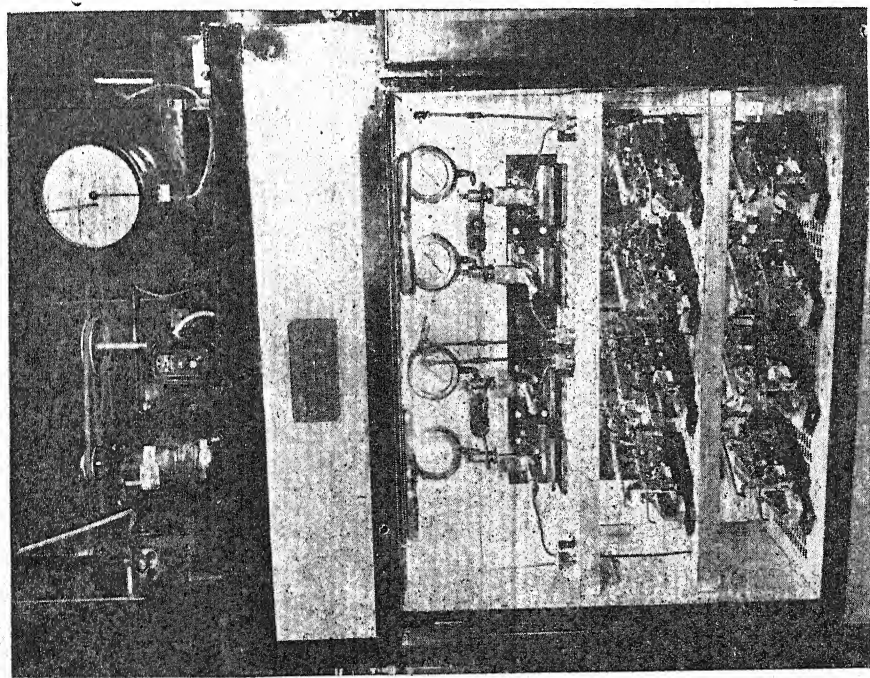


FIG. 3.—Stroking Test Apparatus.

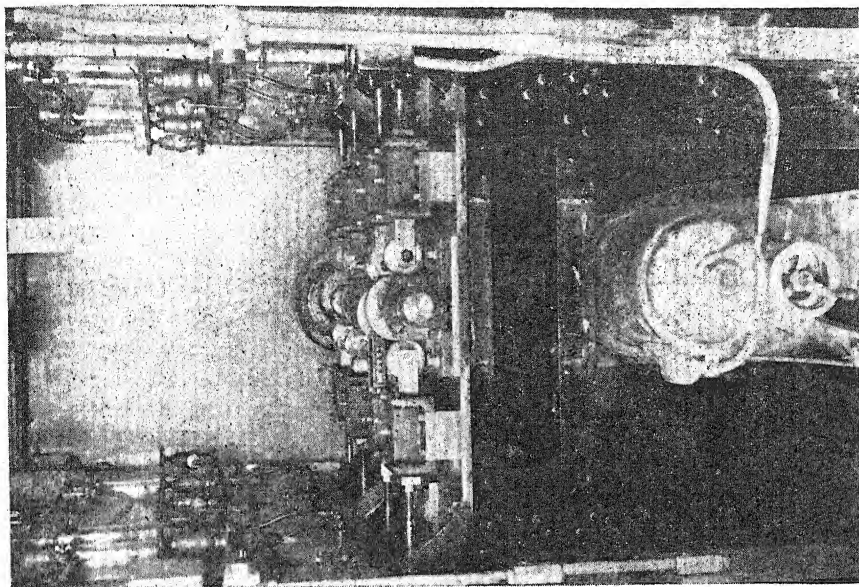


FIG. 4.—Actuating Mechanism for Stroking Test Apparatus.

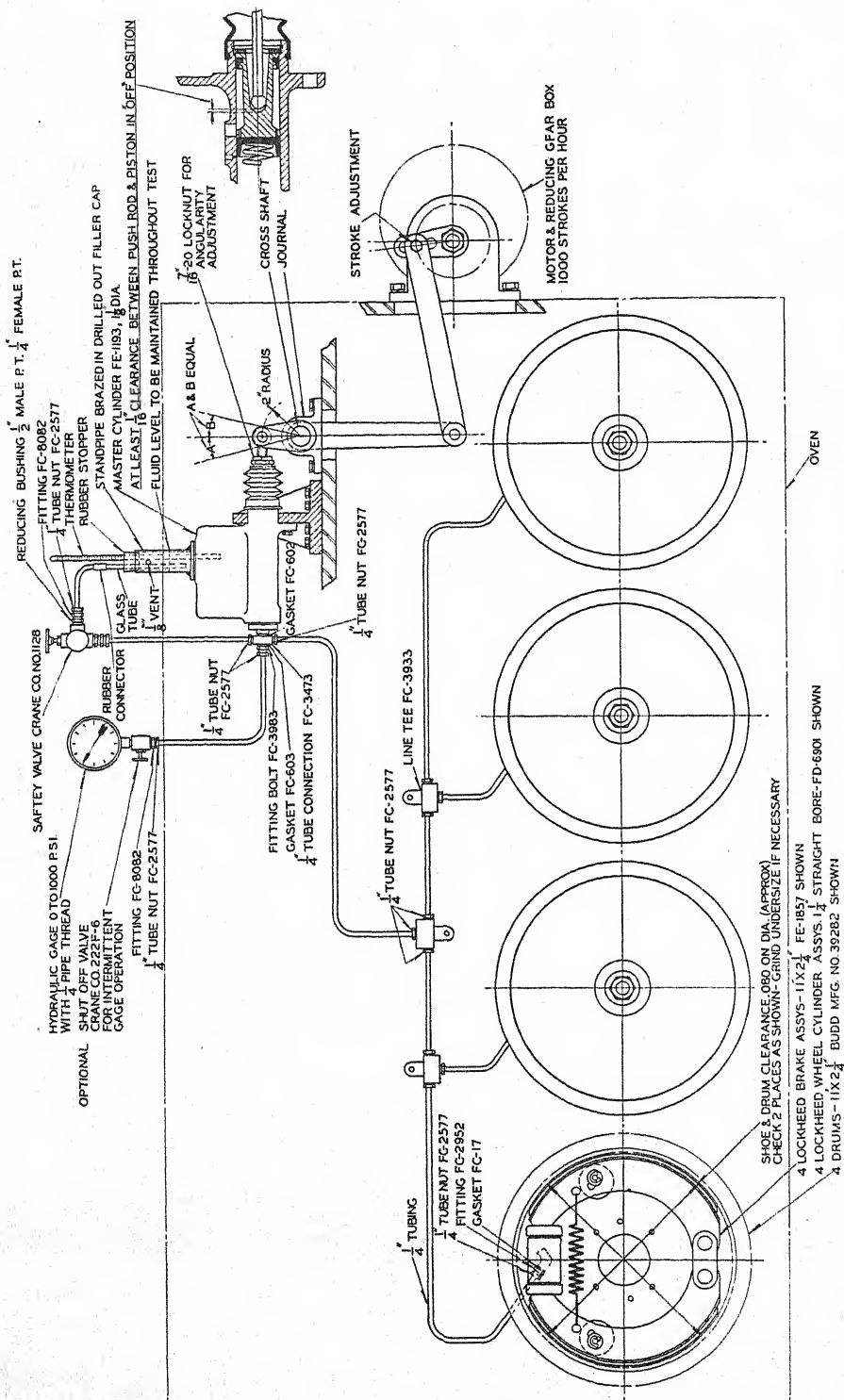


FIG. 5.—Alternative Stroking Test Apparatus.

cylinder  $1\frac{1}{8}$  in. in diameter, four brake assemblies having  $1\frac{1}{4}$  in. diameter wheel cylinders, brake drums, a pressure gage satisfactory for operation at  $500 \pm 50$  psi. and approximately 15 ft. of  $\frac{1}{4}$  in. outside diameter copper or steel tubing shall be used in assembling the apparatus. The master cylinder shall be enclosed in a heated air bath maintained at a temperature of  $158 \pm 2$  F. Mechanical application of pressure to the master cylinder push rod shall be employed to simulate operation on the vehicle as closely as possible. At the start of the test, the brake shoes shall be adjusted concentric with the brake assembly axis to provide a diametral clearance of 0.080 in. between the shoes and drums (0.040 in. clearance on each side). The equipment shown in Fig. 5 has been found satisfactory.

### Test Specimens

22. One primary cup and one secondary cup shall be selected for test specimens (see Section 11 (b)).

### Procedure

23. (a) The specimens shall be wiped with an alcohol-saturated lint-free cloth to remove dirt and packing debris, and then examined and the general condition recorded.

(b) The specimens and master cylinder internal parts shall be dipped in the specified test fluid and the cylinder walls coated with the specified test fluid before assembly.

(c) The durometer hardness of the specimens shall be determined in accordance with Section 30.

(d) The master cylinder (Note) assembly shall be installed in an oven and operated for 150,000 strokes at a rate of  $1000 \pm 100$  strokes per hour and at a temperature  $158 \pm 2$  F. The equipment shown in Figs. 3, 4, and 5 has been found satisfactory.

NOTE.—A master cylinder shall not be used for more than one test.

(e) After this test the specimens shall be removed from the cylinders, wiped with an alcohol-saturated lint-free cloth and cooled for 30 min. on a wooden table top. The durometer hardness shall then be determined in accordance with Section 30 and a thorough inspection made of the cup condition.

### *Low-Temperature Test*

### Apparatus

24. *Cold Chamber.*—The chamber in which the test specimens are exposed to the low temperature shall be of sufficient size to contain the leakage apparatus loaded with specimens and so arranged as to permit the operator to check and operate the apparatus without removal from the chamber. It shall be capable of maintaining a uniform atmosphere of cold dry air within the specified temperature range of  $-40$  to  $-45$  F. The equipment shown in Fig. 6 has been found satisfactory.

### Test Specimens

25. Three wheel cylinder cups, two primary cups, and two secondary master cylinder cups shall be selected as test specimens.

### Procedure

26. (a) The specimens shall be wiped with an alcohol-saturated lint-free cloth to remove dirt and packing debris.

(b) *Hardness Test:*

(1) The durometer hardness shall be determined in accordance with Section 30.

(2) One wheel cylinder cup, one primary cup and one secondary master cylinder cup shall be placed in the cold box for 120 hr. at  $-40$  to  $-45$  F. The durometer and the test jig shall also be subjected to the same temperature for the last  $\frac{1}{2}$  hr. of the 120-hr. period.



While the apparatus is in the cold chamber and the specimen is at a temperature of  $-40$  to  $-45$  F. the durometer hardness shall be determined in accordance with Section 30.

NOTE.—It is necessary to store the durometer used in the cold test in a desiccator for a period of at least 1 hr. before and 1 hr. after use in the cold box to keep it free of moisture.

(c) *Bend Test.*—After an exposure for 120 hr. at  $-40$  to  $-45$  F., the cups shall be bent between the thumb and finger

(2) The specimens shall be wiped clean with an alcohol-saturated lint-free cloth, and assembled in the system, which has previously been conditioned as prescribed in Section 23 (b). The system shall then be filled with the specified test fluid and the air bled from the system.

(3) The complete system shall be enclosed in a cold chamber equipped with a glass door for observation and subjected to a temperature of  $-40$  to  $-45$  F.

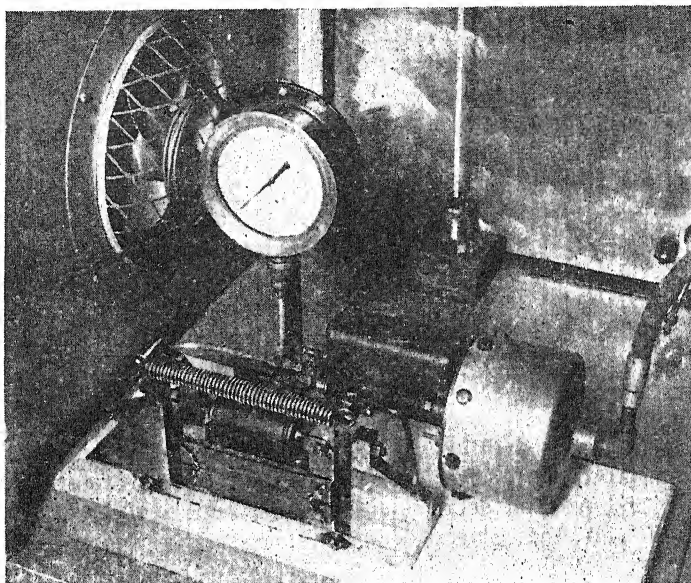


FIG. 6.—Leakage Apparatus Used in Low-Temperature Test.

through an angle of approximately 90 deg. and released immediately. The cold cups shall be bent while in the chamber and shall be handled with gloves to prevent heating from the fingers.

(d) *Leakage Test.*

(1) The apparatus shall consist of a master cylinder and a wheel cylinder so connected that their operation simulates the brake system in actual service. The apparatus shown in Fig. 6 has been found satisfactory.

for a period of 120 hr. During this period no leakage of fluid past the cups shall occur.

(4) The master cylinder piston shall then be operated at an approximate rate of one stroke per minute at a pressure of 500 psi. The piston shall return to the piston stop after each stroke. After 12 piston strokes there shall be no evidence of fluid leakage past the cups.

(5) The operation described in Item (4) shall be repeated at a pressure of 1000 psi.



*Aging Test***Test Specimens**

27. Two cups shall be selected as test specimens.

**Procedure**

28. (a) The specimens shall be wiped with an alcohol-saturated lint-free cloth to remove dirt and packing debris.

min. on a wooden table top to room temperature.

(e) The durometer hardness after aging shall be determined in accordance with Section 30 and the specimens visually inspected.

*Durometer Hardness***Apparatus**

29. (a) *Durometer*.—A durometer,

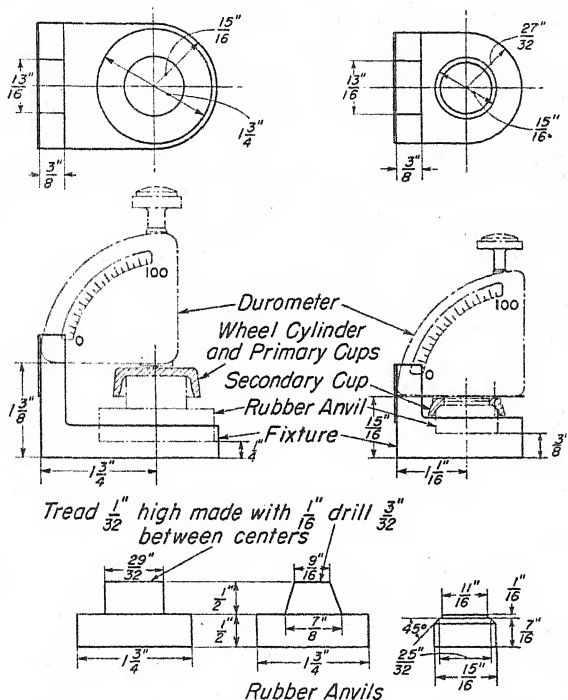


FIG. 7.—Apparatus for Measuring Durometer Hardness.

(b) The original durometer hardness shall be determined in accordance with Section 30.

(c) The two test specimens shall be placed in a circulation air oven as prescribed in A.S.T.M. Method D 573 and held for 70 hr. at  $158 \pm 2$  F.

(d) At the termination of the heating period, the specimens shall be removed from the oven and allowed to cool for 30

type A, which shall be calibrated as described in the Tentative Method of Test for Identification of Rubber by Means of the Durometer (A.S.T.M. Designation: D 676).<sup>3</sup>

(b) *Fixture*.—Fixture, as shown in Fig. 7, for holding the durometer and anvil.

(c) *Anvil*.—Rubber anvil, as shown in

Fig. 7, having a durometer hardness of 46 to 48.

#### Procedure

30. The durometer hardness of the specimen shall be determined using the apparatus as illustrated in Fig. 7. The anvil of the durometer when resting on the specimen shall be level with the indenter point contacting the specimen at the position shown in Fig. 7. The durometer shall not be pressed against

the specimen harder than is necessary to make contact between the rubber and the entire face of the durometer anvil. The immediate reading of the durometer hardness shall be used. The same operator shall make all hardness determinations for any one test.

NOTE.—Since small variations in the pressure of application of the durometer produce considerable variation in results, it is recommended that the above instructions be rigidly adhered to.

# *Tentative Methods of* TESTING RUBBER HOSE<sup>1</sup>



A.S.T.M. Designation: D 380 - 46 T

ISSUED, 1937; REVISED, 1938, 1939, 1940, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods are intended for use in the examination and test of all types and constructions of rubber hose. The term "rubber" as used in these methods includes synthetic compounds as well as compounds of natural rubber.

## General Methods

2. (a) Except as otherwise specified in these methods for rubber hose, the following methods of test of the American Society for Testing Materials, applicable in general to vulcanized rubber, shall be complied with as required and are hereby made a part of these methods:

*General Physical Test Requirements.*—Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15).<sup>3</sup>

*Tension Test.*—Standard Methods

of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412).<sup>3</sup>

*Aging Test.*—Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (A.S.T.M. Designation: D 572)<sup>3</sup> and Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573).<sup>3</sup>

*Adhesion Test.*—Standard Methods of Test for Adhesion of Vulcanized Rubber (Friction Test) (A.S.T.M. Designation: D 413).<sup>3</sup>

(b) In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular hose, the latter shall take precedence.

## Sampling

3. (a) A sample of sufficient length for proper performance of the required tests shall be cut from the hose when possible without impairing its use and the pieces of hose from which samples have thus been cut shall be accepted by the purchaser as full length, provided the hose meets the specified requirements. In general, the amount required

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Latest revision accepted by the Administrative Committee on Standards, September 9, 1946.

Prior to their present publication as tentative these methods were published as tentative from 1933 to 1936. They were adopted in 1936, published as standard from 1936 to 1937, but withdrawn, combined with the Standard Methods of Test for Rubber Hose—Braided Construction (D 379 - 36) and republished as tentative in 1937.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

and the frequency of sampling for each size and type of hose shall be one 30-in. sample from each 500 ft. or fraction thereof of hose over  $1\frac{1}{4}$  in. in nominal diameter, and one 36-in. sample from each 500 ft. or fraction thereof of hose  $1\frac{1}{4}$  in. or under in nominal diameter.

(b) Where the construction of the hose does not permit the cutting of samples as described in Paragraph (a) without impairment of use, as in wire inserted hose, dredging sleeves, etc., a separate length having the same specifications shall be provided, either as an additional length or as a prolongation built on to the hose. Wire shall be omitted from the prolongation or sample length unless the detailed physical tests include requirements that measure the effect of the wire or metal elements in the construction. Additional lengths taken for purpose of tests shall be paid for by the purchaser, provided they conform to the specified requirements.

(c) The sampling of cotton-jacketed rubber-lined hose shall be in accordance with detailed specifications for this material.

#### Measurements of Hose

4. (a) *Length*.—The length shall be determined by means of a steel tape or calibrated measuring device and shall not include the length of fittings except when specifically so requested and recorded. In the case of cotton-jacketed rubber-lined hose, the length shall be determined under a 10-lb. internal pressure.

(b) *Outside Diameter*.—The outside diameter shall be determined by calculation from measurement of the outside circumference. A tape graduated to read the diameter directly may be used.

(c) *Inside Diameter*.—The inside diameter shall be measured with a plug gage except that on hose larger than 3 in.

in nominal diameter, a steel scale or caliper may be used.

#### Thickness of Tube, Cover or Other Rubber Elements

5. (a) The thickness of a rubber element from any type of hose except cotton rubber-lined fire hose shall be determined by means of a standard micrometer graduated to thousandths of an inch and having a presser foot  $0.25 \pm 0.01$  in. in diameter, exerting under dead-weight load a total force of  $3 \pm 0.1$  oz. In the case of cotton rubber-lined fire hose, a spring gage micrometer provided with a presser foot  $0.375 \pm 0.01$  in. in diameter, exerting a total force of 2.1 oz. (60 g.  $\pm 0.1$  oz.) shall be used.

(b) A rubber element adjoining a braid or helical wound reinforcing member of any material shall have the thickness taken as the average between two measurements of thickness, one of which shall be obtained using the unbuffed specimen and the other using a specimen which is buffed just sufficiently to remove the braid or helical corrugations.

(c) A rubber element adjoining a seamless woven jacket of any material shall have the thickness taken on a specimen which is buffed just sufficiently to remove the corrugations caused by the woven jacket. Where backing is used on a rubber element, the backing shall be removed before thickness measurements are taken.

(d) A rubber element adjoining flexible metal convoluted lining shall have the thickness determined at the valleys formed by the tops of the metal convolutions. The opposite side of the rubber element, adjacent to other reinforcing members, shall be prepared for thickness measurement as specified in the other paragraphs of this section, depending upon the type of reinforcing member.

(e) A rubber element adjoining reinforcing fabric woven prior to assembly in the hose, shall have the thickness determined on a specimen which is unbuffed.

#### Test Conditions

6. The temperature of the testing room shall be between 70 and 90 F. (21 and 32 C.). The samples to be tested shall be kept in this room for at least 30 min. previous to the time of testing.

#### Number of Tests

7. (a) When minimum requirements are established, one specimen shall be tested for each physical characteristic required in the detailed specifications for a particular hose except in the immersion tests given in Sections 22 to 25, inclusive. If the results do not meet the specified requirements, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirements, the sample shall be considered to have failed to meet the specifications.

(b) If minimum requirements are not established, at least three specimens shall be tested and the results averaged.

#### TENSILE STRENGTH AND ELONGATION OF RUBBER

##### Apparatus

8. (a) *Testing Machine*.—Tensile strength and elongation tests shall be made on apparatus the error of which does not exceed plus or minus 1 per cent, and provided with an accurate device for indicating the actual maximum load at rupture. The preferred type of apparatus is a power-driven machine of the inclination-balance or pendulum type, but a spring balance accurately calibrated may be used.

(b) *Grips for Testing*.—The grips for holding the test specimen shall be such

that they tighten automatically, exerting a uniform pressure, proportional to the applied tension, across the full width of the enlarged end of the test specimen. The moving grip shall move at a constant speed of 20 in. per min.

#### Test Specimens

9. (a) *Separation of Rubber Parts*.—Sufficient length shall be cut from the hose sample to provide longitudinal tension test specimens from hose  $1\frac{1}{4}$  in. and under in nominal diameter and transverse test specimens on larger sizes. The rubber parts to be tested shall be separated from the balance of the hose without the use of gasoline, if possible. If it is necessary to employ gasoline to effect the separation, it shall be straight run gasoline of the quality known as aviation grade and shall have the following characteristics:

Specific gravity at 60/60 F.... 0.685 to 0.705  
Distillation limits:

Initial boiling point.....	100 to 120 F.
10 per cent evaporated.....	128 to 143 F.
20 per cent evaporated.....	140 to 155 F.
30 per cent evaporated.....	150 to 164 F.
40 per cent evaporated.....	156 to 174 F.
50 per cent evaporated.....	164 to 184 F.
60 per cent evaporated.....	173 to 194 F.
70 per cent evaporated.....	182 to 207 F.
80 per cent evaporated.....	193 to 223 F.
90 per cent evaporated.....	209 to 245 F.
95 per cent evaporated.....	224 to 260 F.
Dry.....	250 to 286 F.
Recovery, min.....	97 per cent

The gasoline shall contain no lead compounds and shall not leave an appreciable amount of oily residue when evaporated.

After use of gasoline, the test specimen shall be allowed to rest at least 1 hr. before being tested.

(b) *Preparation of Specimen*.—The test specimen shall be stamped out from the separated rubber part with dies conforming to Fig. 1 A, B, or C. Die B shall be used when the size of the sample and the tension and stretch limits of the testing machine permit, with exceptions as explained below. If the tension limit is too low for the machine,

Die A shall be used. If the stretch is too high for the machine, Die C shall be used. Die C shall also be used for samples which are too small for Die A or B. In no case shall the thickness of the test specimen exceed  $\frac{1}{8}$  in. or fall below  $\frac{1}{32}$  in. Should it be impossible to obtain test samples having a buffed thickness of  $\frac{1}{32}$  in. or greater, the manufacturer may be requested to furnish a sample of the cured rubber taken from the same lot of rubber as the hose was made from and

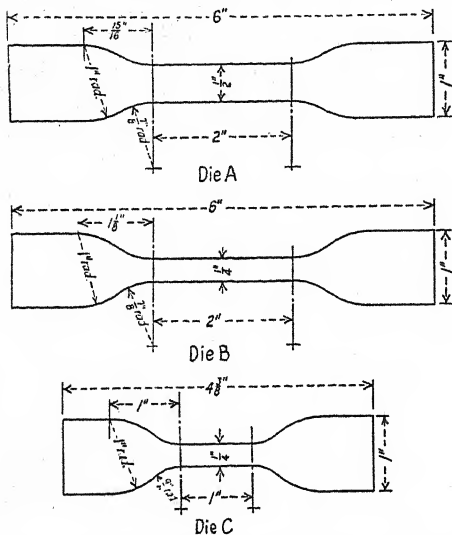


FIG. 1.—Tensile Strength and Elongation Test Specimens.

having a cure equivalent to that to which the hose was subjected. This sample shall be approximately 6 in. in length, and 0.070 to 0.080 in. in thickness, and at least 1 in. in width, and shall be buffed lightly on both sides before the tension test is made. All test specimens, taken either from the hose or from sheets of rubber representing the component parts of the hose, shall be free from marked irregularities of thickness.

(c) *Buffing*.—The specimen shall be buffed, but only to the extent necessary to remove surface corrugations, irregularities, or excess thickness, and to in-

sure smooth faces. Buffing shall be done without excessive heating and the test specimen shall be allowed to rest after buffing for at least 10 min. before being tested.

(d) *Marking*.—Parallel gage lines 1 or 2 in. apart, depending upon the die used, shall be marked on the test specimen at right angles to the longitudinal axis by means of an accurate stamp or bench marker. Care shall be taken to make fine lines and to avoid injuring the specimen.

(e) *Measurement of Test Specimen*.—The thickness of the test specimen shall be the minimum thickness found between the marks on the constricted portion, using the standard micrometer described in Section 5 (a).

### Procedure

10. (a) The test specimen shall be placed in the jaws. The jaws shall be separated at the specified rate of speed until rupture takes place. The distance between the centers of the gage marks noted at the instant of rupture shall be measured using a rule or tape graduated to eighths of an inch or smaller divisions.

(b) Test specimens that break outside of the gage marks or are found to be imperfect shall be discarded and retests shall be made.

### Calculations

11. (a) The tensile strength in pounds per square inch shall be calculated from the breaking load indicated by the machine and the original cross-sectional dimensions of the test specimen.

(b) The elongation shall be the difference between the original gage length and the total distance between the gage marks at the time of rupture as measured in Section 10 (a) and shall be expressed as a percentage of the original gage length.

PERMANENT SET<sup>4</sup>

## Permanent Set Test

12. The specimen for the permanent set test shall be prepared as for determining tensile strength (Section 9) and placed in a suitable stretching device. The grips shall conform to the requirements specified in Section 8 (b). The specimen shall be stretched to an amount equal to three fourths of the ultimate elongation specified, held for 2 min., released, and the distance between the centers of the gage marks measured at the expiration of 2 min. The difference between this measurement and the original gage length divided by the original gage length and expressed as a percentage shall be recorded as the permanent set. If no ultimate elongation is specified, the specimen shall be stretched an amount equal to three fourths of the elongation determined under Section 10.

## ADHESION TESTS

## Test Specimens

13. (a) From cotton-jacketed rubber-lined hose, adhesion test specimens shall be cut transversely from the samples in rings 2 in. in width. These rings shall then be opened at the lap and laid out flat so as to give rectangular specimens of length equal to the full circumference of the hose. On each specimen a central strip of lining  $1\frac{1}{2}$  in. in width shall be cut accurately, the cut extending through the lining but not entirely through the cotton jacket. These strips shall be separated from the jacket at one end for a distance of about  $1\frac{1}{2}$  in.

(b) From other types of hose, the adhesion test specimens shall be cut transversely from the samples in rings 1

in. in width using a sharp tool which will leave clean edges. On hose  $\frac{1}{4}$  to 4 in., inclusive, in internal diameter the specimens shall be tested in ring form and shall be referred to as "ring specimens." For hose over 4 in. in internal diameter the rings shall be cut through at the point where the outside ply of fabric ends, and opened to form strips which shall be referred to as "strip specimens."

(c) Adhesion specimens from hose less than  $\frac{1}{4}$  in. in internal diameter shall be cut longitudinally from the hose. Since the width of these specimens will be less than 1 in., the adhesion shall be determined on the contact width of the test specimen.

(d) In hose constructions having braided wire or woven wire filler reinforcing members, it is impracticable to prepare adhesion test specimens except for determining cover adhesion.

## Procedure

14. (a) *Ring Specimens*.—The ring specimen shall be placed on a mandrel having a diameter equal to the nominal diameter of the hose. The parts to be tested shall be separated sufficiently by hand to permit attaching the jaws of a testing clamp. With the mandrel supported so that it may revolve freely with minimum friction, the prescribed weight shall be attached to the clamp with suitable provision for supporting and releasing it slowly without jerking. The average rate of separation shall not exceed 1 in. per min. The separation shall be noted for a period of 4 min., unless the small size of the specimen makes a shorter period necessary.

(b) *Strip Specimens*.—The various parts to be tested shall be separated at one end of the strip specimen a sufficient distance to permit attaching the jaws of the testing clamp. The strip specimen shall be suspended by the separated end, all separate parts of which, except the

<sup>4</sup> The amount of permanent set is considered by many rubber technologists as having no relation to the service value of rubber compounds except for some specialized requirements. The permanent set test is, therefore, not recommended for general use.



one under test, shall be clamped in the jaws of the testing clamp. A minimum weight sufficient to maintain the strip specimen in approximately a vertical position shall be attached to the lower end of the test specimen, or the specimen may be held against a vertical plate in such a manner as to hold the specimen in approximately a vertical position during the test. The specified weight shall be applied to the separated end with suitable provision for releasing it slowly without jerking. The average rate of separation shall not exceed 1 in. per min. The separation shall be noted for a period of 4 min.

(c) *Report.*—The value of the adhesion shall be reported as average inches of separation per minute under a stated weight.

## HYDROSTATIC TESTS

### Types of Tests

15. Hydrostatic tests of hose consist of subjecting the specimen to the action of internal water pressure under specified conditions to determine either the bursting strength of the hose or the effect of lower hydrostatic pressures in producing changes similar to those occurring in service. In the latter case, the test may be used as an inspection proof test with a minimum safe limit. Bursting strength is determined on short specimens cut from the full hose lengths, but in tests for service effects and for proof inspection, the full lengths are used as specimens. The specimens may be tested, as required, in straight, curved or kinked positions, depending upon the stresses which are desired to be evaluated.

### Application of Hydrostatic Pressure

16. Hydrostatic pressure shall be applied by means of a hand or power-driven hydraulic pump or an accumulator system. The hose shall be connected

to the water line or pump and filled with water prior to application of pressure, allowing all air in the hose to escape through a petcock provided for the purpose. This is important as a safety measure because expansion of air compressed in the hose, when suddenly released by bursting or other failure, might result in serious accident. The petcock shall then be closed and the pressure shall be applied at a uniform rate of increase of approximately 1000 psi. per min., except for hose with bursting strength specified above 2000 psi., in which case the pressure shall be applied at a rate of 10,000 psi. per min. or as rapidly as pump capacity will permit. Pressure shall be measured with a calibrated gage.

NOTE.—The specified rate of 1000 psi. per min. requires too much time for small-diameter high-strength hose, such as hydraulic brake hose, wire reinforced grease gun hose, etc. With  $\frac{1}{8}$ -in. hydraulic brake hose, pressure increases at rates as high as 40,000 psi. per min. are satisfactory.

### Bursting Tests

17. (a) *Straight Bursting Test.*—The specimen for the hydrostatic test for bursting strength shall be at least 18 in. in length for hose 3 in. and smaller in inside diameter, and at least 24 in. in length for larger hose. The pressure shall be raised as specified in Section 16 until failure, and the pressure at which failure occurs shall be considered the bursting strength of the hose.

(b) *Curved Bursting Test.*—The curved bursting strength test for cotton rubber-lined fire hose shall be made in accordance with Paragraph (a), except that the hose shall be firmly secured at both ends to a suitable test frame which is curved to a radius of 27 in.

(c) *Hold Test.*—When a hold test is specified instead of a bursting test, pressure shall be applied as specified in Section 16 until the required pressure is

reached. The specimen shall withstand the specified pressure for a 10-min. period without bursting, leaking, or developing other defects. Specimens which have been subjected to this test shall not be considered fit for service.

### Hydrostatic Pressure Tests

18. (a) When tests for elongation, contraction, twist, warp, rise, and expansion are required, the hose shall be stretched out straight for inspection, and a pressure of 10 psi. applied. All original observations and measurements shall be taken at this pressure. The designated test pressure shall be applied at the rate specified in Section 16 without releasing the original pressure of 10 psi. and final measurements taken. The test pressure shall not be applied longer than 5 sec.

(b) *Proof Pressure Tests.*—When proof pressure tests to determine leakage of hose or couplings are required, the proof pressure shall be applied as in Section 16, and held 1 min. The proof pressure shall not exceed 40 per cent of the specified minimum bursting strength.

(c) *Kink Tests.*—When kink tests are required, each length of hose selected shall be filled with water with the petcock open to allow all air to escape. The petcock shall then be closed and the hose shall be kinked at one end 18 in. from the coupling by tying the coupling back against the hose so that there will be a sharp kink. The pressure shall then be raised as specified in Section 16 to the required pressure and immediately released.

### STEAM TESTS

#### Types of Test

19. (a) Steam tests of hose consist of subjecting test specimens having previously determined physical properties to the action of steam under controlled

conditions for known periods after which the physical properties are again measured and the changes noted. Two types of test that differ in the manner in which the specimens are exposed to the action of the steam are covered, as follows:

*Rack Method.*—The steam is in contact only with the interior of the hose.

*Digester Method.*—The specimen is entirely surrounded by steam.

The choice of type of test to be used for a given hose sample depends on the kind of service being evaluated.

(b) Tensile strength and ultimate elongation of the rubber parts and strength of adhesion are the physical properties used in addition to visual and manual inspection for evaluating the effect of the steam. These shall be determined in accordance with Sections 8 to 11, inclusive, and Sections 13 and 14.

#### Rack Method

20. (a) *Apparatus.*—Two fixed horizontal steam headers having suitable connections for attaching specimens shall be placed one above the other at such distance that the specimens will just fit between the connections in a vertical position without distortion. Dry saturated steam at the required pressure shall be supplied to the specimens through the upper header which shall be equipped with a pressure regulating valve, a recording gage, and suitable indicating gages. The lower header shall be connected to a steam trap. Shutoff valves shall be provided at each opening in each header.

(b) *Test Specimen.*—The test specimen shall be a piece of the hose sample cut to length as required by the apparatus except that the length shall be not less than 16 nor more than 24 in.

(c) *Procedure.*—For determining the physical properties before steaming, a portion of the hose sample not required for the rack test shall be used. The

specimen for steaming shall be mounted in the apparatus and subjected to internal steam pressure of  $75 \pm 2$  psi. for 7 hr. on each of two successive days, the steam being turned off and the hose allowed to cool during the 17-hr. intermediate interval. After the second steaming period, the hose shall be removed from the apparatus, allowed to cool, and held under laboratory atmospheric conditions for not less than 12 hr. nor more than 48 hr. after which the physical properties after steaming shall be determined.

### Digester Method

21. (a) *Apparatus*.—A suitable pressure vessel having a steam inlet with a regulating valve and a condensate outlet through a steam trap shall be provided. The vessel shall have a petcock air vent for escape of trapped air at the beginning of the test and shall be equipped with a recording thermometer or pressure gage. A perforated metal or wire frame shall be provided for holding the specimens at least 6 in. above the condensate outlet.

(b) *Test Specimen*.—The test specimen shall be a piece of the hose sample cut to such length that all test specimens for physical properties can be obtained after discarding at least 2 in. at each end.

(c) *Procedure*.—For determining the physical properties before steaming, a portion of the hose sample not required for the digester test shall be used. The specimen for steaming shall be placed in the digester and surrounded by dry saturated steam at a pressure of  $45 \pm 3$  psi. for 48 hr. continuously. At the conclusion of this period, the pressure in the digester shall be released, the hose removed and examination made at once for blistering of the inner tube or cover or loosening of the tube from the fabric. The specimen shall be allowed to cool and to rest under laboratory atmospheric conditions not less than 12 nor more than

48 hr. after which the physical properties after steaming shall be determined.

### IMMERSION TESTS FOR SWELL AND DETERIORATION OF HOSE USED FOR PETROLEUM PRODUCTS

#### Types of Test

22. Immersion tests for evaluating the swelling and deteriorating effects of petroleum products on rubber hose consist of subjecting suitable test specimens from the rubber portion having known physical properties to the action of selected solvents by immersing the specimens completely in solvent under controlled conditions for definite periods, after which the physical properties are again determined and the changes noted. Two types of test are covered, as follows:

*Swelling Test*.—This type involves measurement of the change in volume of the rubber caused by the swelling action of the solvent.

*Strength Deterioration Test*.—In this type the change in strength is measured. Alternative procedures using different solvents are given.

In any of the immersion tests, the choice of method and solvent depends upon the nature of the actual service and the petroleum product which is there being used.

#### Gasoline Immersion Swelling Test

23. (a) *Test Specimens*.—The test specimens shall be rectangular rubber blocks 2 by 1 in. having a thickness not over  $\frac{1}{16}$  in. They shall be cut from the hose and buffed on both faces only to the extent necessary to insure smoothly buffed faces, except when the material is too thick, in which case the buffing shall be sufficient to reduce the specimens to  $\frac{1}{16}$  in. Three specimens shall be used for each test and the results averaged.

(b) *Procedure*.—The volume of each test specimen shall be measured by the water displacement method in which the

specimen is accurately weighed to the nearest milligram in air ( $W_1$ ) and in distilled water ( $W_2$ ) at room temperature. When weighing in water, care shall be taken that the specimen is free from adhering air bubbles and, if necessary, it may first be wetted by being dipped in 95 per cent ethyl alcohol and thoroughly rinsed with distilled water. After weighing, the specimen shall be blotted dry with filter paper, completely immersed in gasoline, and allowed to stand for 24 hr. at room temperature. The gasoline used shall be straight run aviation grade as described in Section 9 (a). Excessive evaporation may be avoided by fitting the container loosely with a cork stopper. At the termination of the immersion period, the specimen shall be removed from the gasoline, dipped in 95 per cent ethyl alcohol, blotted lightly with filter paper, and placed in a tared weighing bottle and weighed ( $W_3$ ). It shall then be removed from the bottle and weighed ( $W_4$ ) in distilled water in immediate consecutive procedure to determine the water displacement after test. The final weighing shall be completed within 5 min. after removal from the gasoline.

(c) *Calculation.*—The percentage increase in volume shall be calculated as follows:

Percentage increase in volume =

$$\frac{(W_3 - W_4) - (W_1 - W_2)}{(W_1 - W_2)} \times 100$$

#### Kerosine-Benzol Immersion Strength Deterioration Test

24. (a) *Test Specimens.*—Six dumbbell-shaped test specimens, prepared as described in Section 9, will be required. Both faces of the dumbbell ends shall be buffed.

(b) *Apparatus.*—The apparatus, in which the specimen is subjected to the

action of the solvent, shall consist of a glass container fitted tightly with a cork stopper and an efficient reflux condenser. The specimen shall be supported at least  $\frac{1}{2}$  in. from the bottom of the tube by means of a wire screen or similar device.

(c) *Kerosine.*—The kerosine shall be of a grade conforming to the following requirements:

Test	A.S.T.M. Method <sup>a</sup>	Limit
Burning quality test, hr.....	D 187	16 min.
Flash point, deg. Fahr.....	D 56	115 min.
End point, deg. Cent.....	D 86	330 max.
Cloud point, deg. Fahr.....	D 97	5 max.
Sulfur, per cent.....	D 90	0.13 max.
Color, Saybolt number.....	D 156	+16 min.

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Burning Quality of Kerosine Oils (A.S.T.M. Designation: D 187),<sup>5</sup>

Standard Method of Test for Flash Point by Means of the Tag Closed Tester (A.S.T.M. Designation: D 56),<sup>5</sup>

Standard Method of Test for Distillation of Gasoline, Naphtha, Kerosine, and Similar Petroleum Products (A.S.T.M. Designation: D 86),<sup>5</sup>

Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97),<sup>5</sup>

Tentative Method of Test for Sulfur in Petroleum Oils by Lamp Method (A.S.T.M. Designation: D 90),<sup>5</sup> and

Standard Method of Test for Color of Refined Petroleum Oil by Means of Saybolt Chromometer (A.S.T.M. Designation: D 156).<sup>5</sup>

(d) *Benzol.*—The benzol shall be a pure, water white grade, free from suspended matter and conforming to the following requirements:

Specific gravity at 60/60 F.... 0.875 to 0.886  
Distillation limits:

Initial boiling point..... 168 to 172 F.

95 per cent evaporated... 174 F., max.

Dry..... 176 F., max.

Recovery..... 98 per cent, min.

(e) *Procedure.*—Three specimens shall be tested for tensile strength and elongation in accordance with Section 10 and the results averaged to give the properties of the rubber before immersion. The remaining three specimens shall be placed in the immersion apparatus and completely surrounded by solvent consisting of 85 per cent kerosine and 15 per cent benzol. With the reflux condenser in place the apparatus containing

the specimen shall then be maintained at a temperature of 135 to 140 F. (57 to 60 C.) for 48 hr. Use of a thermostatically controlled liquid bath is recommended. At the end of this period, the specimens shall be removed, dipped once in acetone at room temperature, and blotted lightly with filter paper. The specimens shall then be suspended or placed on a screen and allowed to dry at room temperature in air protected from drafts for 4 hr., after which the tensile strength and elongation after immersion shall be determined as before, except that the measurements for cross-sectional area and test length between bench marks shall be taken after immersion and drying. The deterioration shown by the difference between the determinations before and after immersion shall be expressed as a percentage of the original property.

#### Oil Immersion Strength Deterioration Test

25. This determination shall be made exactly as in the case of the kerosine-benzol test described in Section 24, except that the temperature during immersion shall be maintained at  $200 \pm 2$  F. ( $93 \pm 1$  C.) for 48 hr. and the solvent shall be a neutral, pale, paraffin-base oil. The oil shall be yellow in color with blue fluorescence, free from turbidity, and shall conform to the following requirements:

Test	A.S.T.M. Method <sup>a</sup>	Minimum	Maximum
Specific gravity, per cent. ....	.....	0.878	0.882
Flash point, deg. Fahr. ....	D 92	330	340
Saybolt Universal Viscosity at 122 F., sec. ....	D 88	61	66

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92),<sup>6</sup>

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>6</sup>

### INSPECTION AND REJECTION

#### Inspection

26. (a) All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. The manufacturer shall afford the inspector, without charge, all reasonable facilities to test and inspect the hose.

(b) The purchaser may make the tests and inspection to govern the acceptance or rejection of the material in his own laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser not later than 60 days after receipt of the material.

#### Retests and Rejection

27. (a) Any material that fails in one or more tests may be resampled and retested at the expense of the manufacturer. For this purpose two additional samples shall be selected from the hose for this test that failed to meet the requirements. Failure of either of the retested samples shall be cause for final rejection.

(b) Rejected material shall be disposed of as directed by the manufacturer and at his expense.

# *Tentative Methods of*

## TESTING AUTOMOTIVE AIR BRAKE AND VACUUM BRAKE HOSE<sup>1</sup>



A.S.T.M. Designation: D 622 - 44 T

ISSUED, 1941; REVISED, 1944.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods are intended for use in testing the conventional types or hose employed for the operation of air brake and vacuum brake systems, either on a single motor vehicle or as connecting or transmission lines in a combination of vehicles. The hose may be assembled with suitable metal end fittings or may be as fabricated for use with detachable fittings. The term "rubber" as used in these methods includes synthetic compounds as well as compounds of natural rubber. These methods do not include determinations of tensile strength and elongation of rubber compounds, since the characteristics of these are considered to be adequately covered by the other physical tests.

### General Methods

2. (a) The special tests described in these methods shall be applied as re-

quired by detailed specifications for these types of hose. Otherwise, the general methods in the Tentative Methods of Testing Rubber Hose (A.S.T.M. Designation: D 380) of the American Society for Testing Materials<sup>3</sup> shall be used and are hereby made a part of these methods.

(b) In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular hose, the latter shall take precedence.

### Sampling

3. (a) One representative sample of each lot to be tested shall be taken. The total length of hose required for all tests is as follows:

Air brake hose.....approximately 6 ft.  
Vacuum brake hose.....approximately 10 ft.

(b) Each test specimen shall be prepared from the original sample without

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

having been subjected to any previous test.

(c) In the interest of safety, any hose remaining intact after these tests shall be destroyed by cutting through at the center of the length.

### Test Conditions

4. The temperature of the testing room shall be maintained between 70 and 90 F. (21 and 32 C.). The test samples shall be held at room temperature prior to testing until their temperature is stabilized.

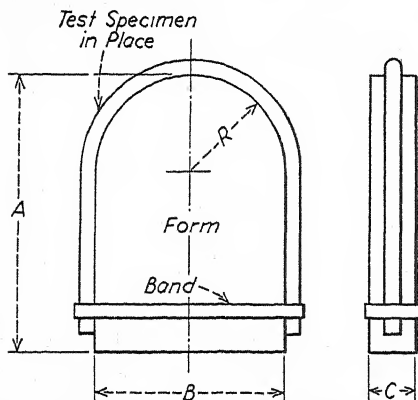


FIG. 1.—Test Specimen on Form for Aging Test (see Table I).

### Air Pressure Test

5. A specimen of hose 18 in. in length, assembled with end fittings as for service, shall be connected to a source of air pressure. The hose and couplings shall then be entirely submerged in water, using any suitable container for the water such that visual observation of the assembly is permitted. Air pressure of 200 psi. shall then be applied internally and maintained for 5 min. Any evidence of leakage of air from the hose or couplings shall be considered failure to meet this test.

### Bursting Test

6. The bursting test shall be con-

ducted in accordance with Section 17 (a) of the Tentative Methods of Testing Rubber Hose (A.S.T.M. Designation: D 380) of the American Society for Testing Materials.<sup>3</sup>

### Aging Test

7. A specimen of hose of the length prescribed in Table I shall be bent around a form of the dimensions speci-

TABLE I.—DIMENSIONS OF TEST SPECIMEN AND FORM FOR AGING TEST.

Inside Diam- eter of Hose, in.	Length of Specimen, in.	Dimensions of Form, in. (see Fig. 1)			
		A	B	R	C (min.)
AIR BRAKE HOSE					
1/4	9	4 1/2	3	1 1/2	1 1/2
3/8	10	4 3/4	3 1/2	1 3/4	1 3/4
1/2	10	4 3/4	3 1/2	1 3/4	1 3/4
3/4	11	5	4	2	1 3/4
1	11	5	4	2	1 3/4
1 1/4	12	5 1/2	4 1/2	2 1/4	1 3/4
1 1/2, special	14	6	5	2 1/2	1 3/4
VACUUM BRAKE HOSE					
1/4	9	4 1/2	3	1 1/2	1 1/2
3/8	10	4 3/4	3 1/2	1 3/4	1 3/4
1/2	11	5	4	2	1 3/4
3/4	14	6	5	2 1/2	1 3/4
1	16	7	6 1/2	3 1/4	1 3/4

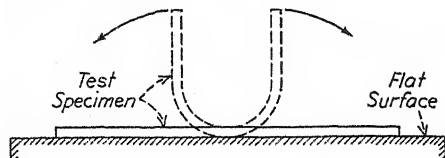


FIG. 2.—Aged Specimen Being Straightened.

fied in Table I and held in place by a band or cord (see Fig. 1). The assembly shall be placed for 4 days at 194 F. (90 C.) in an air oven conforming to that described in the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573) of the American Society for Testing Materials.<sup>3</sup> After removal from the oven, the hose shall be allowed to cool to room temperature and shall then be removed from the form. It shall then be opened out to a straight length, as shown in Fig. 2, and



examined externally for cracks, charring, or disintegration. The specimen shall be cut lengthwise and the inner tube shall be examined for signs of cracking.

#### Cold Test

8. The hose shall be conditioned in a cold box in a straight position at  $-40^{\circ}\text{F}$ . ( $-40^{\circ}\text{C}$ .) for 72 hr. After conditioning and without removal from the cold box, the hose shall be bent around a mandrel having a diameter ten times the outside diameter of the hose, and shall not break nor crack.

#### Air Brake Hose

##### Tensile Strength Test

9. A tension testing machine conforming to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for Testing Materials<sup>3</sup> shall be used. The machine shall be provided with a recording device to give the total pull in pounds at the conclusion of the test and shall be operated at a speed of approximately 1 in. per min. The specimen of air brake hose, approximately 18 in. in length, shall be so held in the testing machine that the hose and fittings have a straight center line corresponding to the direction of the machine pull. The hose assembly shall be subjected to an increasing tension load until failure occurs, either by separation of the specimen from the end fittings or by rupture of the hose structure. Notation shall be made as to the type of failure.

##### Swelling Test

10. The swelling test on air brake hose shall be conducted in accordance with Section 23 of the Tentative Methods of Testing Rubber Hose (A.S.T.M. Designation: D 380),<sup>3</sup> except that A.S.T.M. precipitation naphtha<sup>4</sup> shall be used as the immersion medium. The test

specimen shall be prepared from the hose tube and cover as described in Section 9 (a) of Methods D 380 and shall be as near the size specified in Section 23 (a) of Methods D 380 as can be obtained from the hose being tested.

#### Vacuum Brake Hose

##### Bend Test

11. A specimen of vacuum brake hose of the length prescribed in Table II shall be bent in the direction of its normal curvature until its ends just touch as shown in Fig. 3. The outside diameter of the specimen at the middle section A shall be measured in the plane of the center line before and after bending,

TABLE II.—DIMENSIONS OF BEND TEST SPECIMEN OF VACUUM BRAKE HOSE.

Inside Diameter of Hose, in.	Length of Specimen, in.
$\frac{1}{4}$	8
$\frac{3}{8}$	12
$\frac{1}{2}$	16
$\frac{3}{4}$	28
1	36

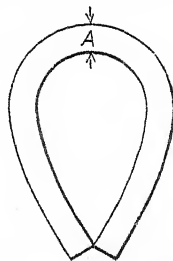


FIG. 3.—Bend Test of Vacuum Brake Hose.

using outside spring calipers and a steel scale graduated to  $\frac{1}{64}$  in. The difference between the two measurements shall be considered the collapse of the hose outside diameter on bending.

##### Vacuum Test

12. A specimen of vacuum brake hose 12 in. in length shall be assembled with

<sup>4</sup> Requirements for this precipitation naphtha are given in Section 4 of the Standard Methods of Test for Precipitation Number of Lubricating Oils (A.S.T.M. Designation: D 91), 1946 Book of A.S.T.M. Standards, Part III-A.

suitable end fittings such that one end may be completely closed against air leakage and the other end connected to a vacuum pump. The outside diameter of the test specimen shall be measured, and the specimen shall then be subjected to a vacuum of 26 in. of mercury for 5 min. A suitable manometer or vacuum gage shall be connected in the system to indicate the degree of vacuum actually maintained. At the end of the 5-min. period, while the hose is still under vacuum, the outside diameter of the

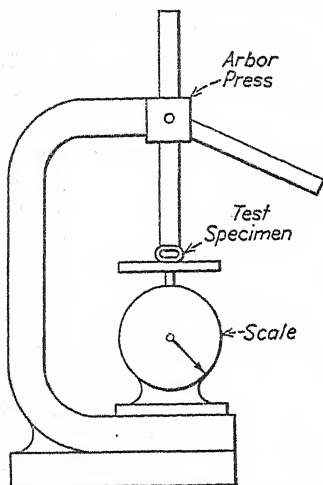


FIG. 4.—Apparatus Assembly for Deformation Test of Vacuum Brake Hose.

specimen shall again be measured so as to determine the minimum diameter at any cross-section. The measurement shall be made using outside spring calipers and a steel scale graduated to  $\frac{1}{64}$  in. The difference between this measurement and the original outside diameter shall be considered the collapse of the hose outside diameter under vacuum.

#### Deformation Test

13. (a) *Apparatus*.—The apparatus for the deformation test of vacuum brake hose shall consist essentially of a No. 3 arbor press or other suitable compression

device (Fig. 4) for collapsing the diameters of hose specimens, a platform scale or other suitable means for weighing the load required to collapse the hose, and feeler gages for measuring the free distance between the inner tube faces of the collapsed hose ( $D$  in Fig. 5). The weighing device shall have a capacity of at least 100 lb. and shall indicate the applied loads on a dial or scale with an accuracy within plus or minus 1 per cent. The feeler gages shall be of sufficient length to be passed completely through specimens of hose 1 in. in length, and shall be

TABLE III.—DIMENSIONS OF TEST SPECIMEN AND FEELER GAGE FOR DEFORMATION TEST OF VACUUM BRAKE HOSE.

Inside Diameter of Hose, in.	Specimen Dimensions, in. (see Fig. 5)		Feeler Gage Dimensions, in.	
	$D$	$L$	Width	Thickness
$\frac{1}{4}$ .....	$\frac{1}{16}$	1	$\frac{1}{8}$	$\frac{1}{16}$
$\frac{3}{8}$ .....	$\frac{3}{32}$	1	$\frac{3}{16}$	$\frac{3}{32}$
$\frac{1}{2}$ .....	$\frac{1}{8}$	1	$\frac{1}{4}$	$\frac{1}{8}$
$\frac{3}{4}$ .....	$\frac{3}{16}$	1	$\frac{3}{8}$	$\frac{3}{16}$
1.....	$\frac{1}{4}$	1	$\frac{1}{2}$	$\frac{1}{4}$

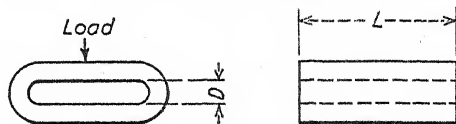


FIG. 5.—Deformed Specimen of Vacuum Brake Hose (see Table III).

of rectangular cross-section with dimensions as prescribed in Table III.

(b) *Test Specimen*.—The test specimen shall consist of a section of the vacuum brake hose cut to a length of 1 in.

(c) *Procedure*.—The test specimen shall be placed longitudinally in the arbor press and compressed five consecutive times to the form shown in Fig. 5, with dimension  $D$  as prescribed in Table III. The specimen shall be held under load each time for 5 sec. and shall be allowed to recover for approximately 10 sec. which shall elapse between load applications. While under each application of

the load dimension  $D$  shall be measured by means of the proper feeler gage for the size of hose being tested. Between the fourth and fifth load application, the weighing device shall be inserted in the press and the specimen placed on it so that the fifth load application may be measured. The observed load required in the fifth application to compress the specimen to the specified dimension  $D$  shall be recorded. The specimen shall be considered to have failed to meet the test unless this load exceeds 40 lb. If desired, the test may be made with the weighing device in the press during all load applications, but in this case, a scale capacity substantially greater than 100 lb. should be provided to avoid damaging the scale during the first load applications.

#### Swelling Test

14. (a) A specimen of vacuum brake hose 12 in. in length shall be filled with A.S.T.M. precipitation naphtha<sup>4</sup> enclosed by means of suitable cork stoppers to prevent loss by evaporation or leakage. Use of shellac on the stoppers and joints is an aid in obtaining the proper seal. Care shall be taken in sealing the hose to avoid putting the naphtha under greater than atmospheric pressure. The filled hose shall be allowed to stand at room temperature for 48 hr., after which the naphtha shall be removed and the hose specimen shall be

immediately subjected in sequence to the tests described in Paragraphs (b) to (d).

(b) A steel ball of specified diameter shall be dropped through the specimen. If the ball does not pass freely, the specimen shall be recorded as having failed to meet the test.

(c) The specimen shall next be subjected to a vacuum of 26 in. of mercury as prescribed in Section 12, except that no measurements of diameter are required and the vacuum shall be maintained for 10 min. Leakage of air through the hose shall be recorded as failure to meet the test.

(d) The specimen shall then be cut lengthwise in two sections and examined for any signs of separation of the inner tube from the fabric. Such separation shall be recorded as failure to meet the test.

#### REPORT

##### Report

15. The report shall include the following:

(1) Description of the sample including type and size of hose and type of couplings, if any,

(2) Statement of the methods used together with the results obtained,

(3) All observed and recorded data, and

(4) Date of test, and temperature of test room.

# *Tentative Methods of*

## TESTING AUTOMOTIVE HYDRAULIC BRAKE HOSE<sup>1</sup>



A.S.T.M. Designation: D 571 - 44 T

ISSUED, 1940; REVISED, 1944.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods are intended for use in testing assemblies of  $\frac{1}{8}$ -in. hydraulic brake hose with end fittings as used in the automotive industry, and include procedures for determining volumetric expansion under pressure, bursting strength, fatigue life, and tensile strength.

### Test Specimens

2. The test specimens shall consist of coupled sections of  $\frac{1}{8}$ -in. hydraulic brake hose having a measured free length of hose exposed between the end fittings. The free lengths of specimens to be used in the whip test for fatigue life shall be not less than 9 in. nor more than  $15\frac{1}{2}$  in.

### Test Conditions

3. The temperature of the testing room shall be maintained between 70 and 90 F. (21 and 32 C.). The specimens shall be stabilized at the testing room temperature prior to testing.

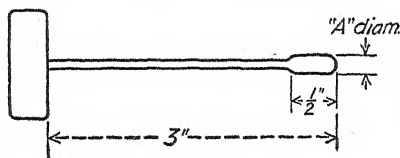
<sup>1</sup>Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup>Revisions accepted by the Society in June, 1944, and Committee E-10 on Standards, December 13, 1944.

### MEASUREMENTS OF HOSE

#### Procedure

4. (a) Constriction of the bore after assembly of the hose and fittings shall be measured with a gage plug as shown in the accompanying Fig. 1 with dimension "A" as prescribed in the detailed specifications for the hose. The gage plug shall weigh  $2 \pm 0.1$  oz. The hose



Gage plug shall weigh  $2 \pm 0.1$  oz. Dimension "A" of the gage plug shall be as prescribed in the specifications for the hose.

FIG. 1.—Gage Plug for Testing Constricting of Bore of Hose.

assembly shall be held in a vertical position and the "A" diameter portion inserted into the end of the fitting. The gage shall be permitted to drop, of its own weight, a distance of 3 in. into the hose assembly. The time required to drop 3 in. shall not exceed 5 sec.

(b) Length of hose assembly shall be measured with the assembly in a vertical position with a weight of  $1.25 \text{ lb.} \pm 0.1$

oz. attached to one end. A vernier caliper scale or equivalent shall be used and the length reported in inches and thousandths of an inch.

## EXPANSION TEST

### Description

5. This expansion test is designed to measure, by fluid displacement, the volumetric expansion of the free length of assembled hydraulic brake hose when subjected to specified internal pressures. The free length shall be the length between the fittings measured as described in Section 4 (b).

### Apparatus

6. The test apparatus shall consist essentially of the following: A suitable source for the required fluid pressures, pressure gages, piping, valves, fittings in which the hose assembly may be mounted in a vertical position for application of pressure under controlled conditions, and a graduated burette for measuring the volume of liquid corresponding to the expansion of the hose under pressure. All piping and connections shall be smoothbore without recesses or offsets, so that all air may be freely removed from the system before running each test. The valves shall be of such design as to open and close with minimum displacement of liquid.<sup>3</sup> The apparatus shall be capable of applying the pressure at a rate of increase of  $25,000 \pm 10,000$  psi. per min. A suitable apparatus is illustrated in Fig. 2.

### Calibration of Apparatus

7. The apparatus shall be tested prior to use to determine its calibration correction factor. For this purpose,  $\frac{1}{4}$ -in. diameter Bundy steel tubing or its

equivalent shall be used in place of the hose assembly. Calibration correction factors shall be established at pressures of 1000 and 1500 psi., and these shall be subtracted from the expansion readings obtained on the test specimens. The maximum permissible calibration correction factor shall be 0.08 cu. cm. at 1500 psi.

### Procedure

8. The test specimen shall be carefully threaded into position on the apparatus in such a way as to provide a leak-proof seal, care being taken to avoid twisting, and to maintain the hose in a vertical position without tension while under pressure. The tank *C* shall be filled with distilled water, taking care that it is free of air or dissolved gases. Valve *B* shall then be opened and crank *D* turned to the left to allow the maximum amount of water to flow into the master cylinder. Valves *E*, *F*, and *G* shall next be opened, allowing the water to run from tank *C* through burette *K* until no air bubbles are seen in the burette. Removal of air bubbles may be facilitated by moving the hose back and forth. Valves *B* and *F* shall then be closed and the pressure in the hose raised to 1500 psi. for not more than 10 sec. After inspection for leaks at the connections, the pressure in the hose shall be completely released by opening valve *F*, which shall then be closed before proceeding with the next step. The water level in burette *K* shall now be adjusted to zero by means of valve *G*. Crank *D* shall then be turned to the right until gage *A* shows a pressure of 1000 psi. This pressure shall be sealed in the hose by closing valve *E*, after which the expansion shall be taken immediately by opening valve *F* and allowing the water in the expanded hose to rise in the burette. As soon as the liquid level is constant, valve *F* shall be closed and

<sup>3</sup> Crane Globe Valve No. 222X, or equivalent has been found satisfactory.

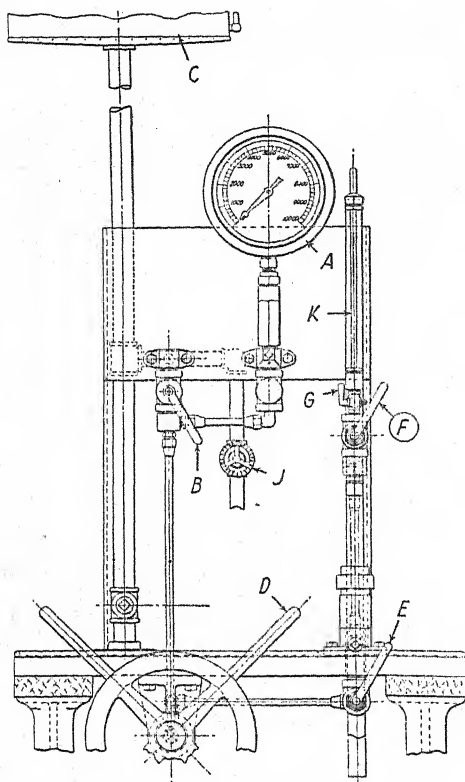
the reading taken on burette *K*. This operation shall be repeated so that the final reading taken on burette *K* will be the total of three expansions. This reading divided by three, minus the calibration factor shall be considered as the final volumetric expansion of the

reading shall be taken, but instead, the pressure shall be completely released and the procedure repeated.

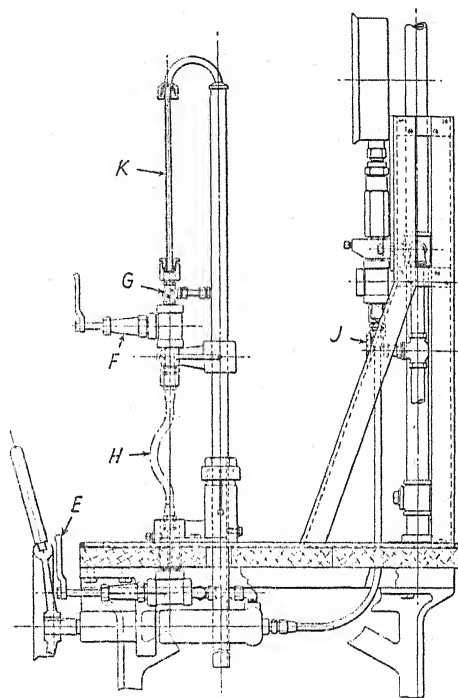
#### BURSTING STRENGTH TEST

##### Description

9. This bursting strength test con-



A—Pressure gage of 10,000-psi. capacity.  
B—Control valve from tank C.  
C—Pressure-medium tank (vented on top).  
D—Screw operated master cylinder.  
E—Valve controlling line from master cylinder to hose.



F—Valve above hose.  
G—Valve controlling flow into burette.  
H—Hose in slack position.  
J—Fluid line valve.  
K—Burette graduated in 0.1 cu. cm.

FIG. 2.—Apparatus for Expansion and Bursting Strength Tests.

hose at 1000 psi. The water level in the burette shall then be readjusted to zero as above and the procedure repeated to obtain the expansion at a pressure of 1500 psi. If the pressure in the hose should inadvertently be raised just prior to the expansion reading to a value above that specified, no

sists of subjecting the specimen of hydraulic brake hose to the action of internal hydrostatic pressure under specified conditions.

##### Apparatus

10. The apparatus shall consist of a suitable pressure system in which the

hose is so connected that controlled and measured fluid pressure may be applied internally. The pressure shall be obtained by means of a hand or power-driven pump or an accumulator system and shall be measured with a calibrated gage. Provision shall be made for filling the hose with distilled water and allowing all air to escape through a relief valve prior to application of pressure. This is important as a

to the pressure system and completely filled with water, allowing all air to escape. Removal of air bubbles may be facilitated by moving the hose back and forth. The relief valve shall then be closed and the pressure shall be applied at the prescribed rate until it reaches 3000 psi., which shall be held for 1 min. At the expiration of this "hold" period, the pressure shall be increased to 4000 psi. and again maintained for 1

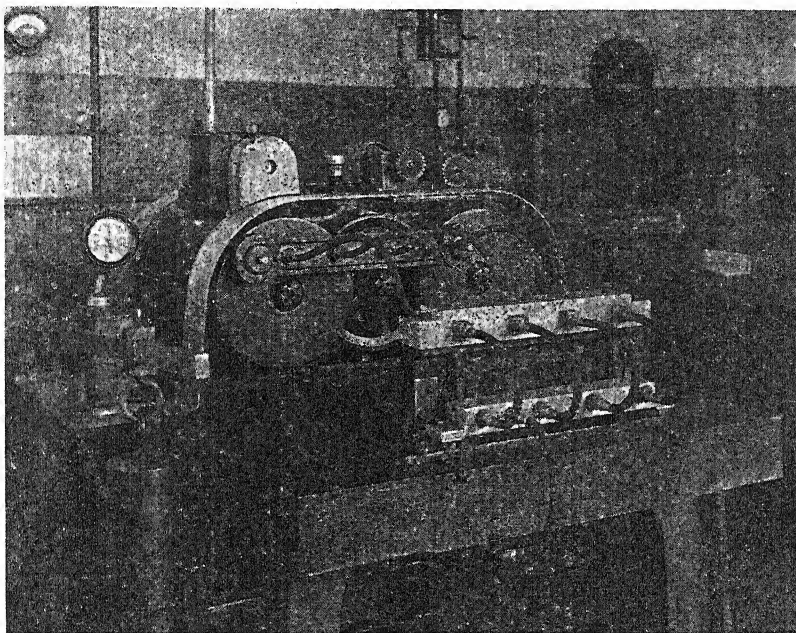


FIG. 3.—Whip Test Apparatus for Determining Fatigue Life.

safety measure. The pressures shall be applied at a rate of increase of  $25,000 \pm 10,000$  psi. per min. Since this type of hose withstands a minimum bursting pressure of 5000 psi., care must be taken that all piping, valves, and fittings are sufficiently rugged and adapted to high-pressure work. The apparatus described for the expansion test may be used when it conforms to these requirements.

#### Procedure

11. The specimen shall be connected

min., after which it shall be increased until the specimen bursts. The maximum pressure noted on the calibrated gage shall be considered as the bursting strength of the specimen.

#### FATIGUE LIFE (WHIP TEST)

##### Description

12. This whip test is designed to measure the fatigue life of the brake hose assembly. The flexing motion imparted by the test apparatus, while



different from that to which the assembly is subjected in service, provides a highly accelerated method of measuring the resistance of a hose to dynamic fatigue.

#### Apparatus

13. The essential features of the apparatus, illustrated in Fig. 3, are as follows: A moveable header consisting of a horizontal bar mounted at each end on vertically rotating disks through ball bearings with centers placed 4 in. from the disk centers, and an adjustable stationary header parallel to the moveable header in the same horizontal plane as the centers of the disks. The headers are each provided with four standard end connections equally spaced, approximately  $3\frac{1}{2}$  in. on centers in which the hose assemblies are mounted in parallel. The disks are revolved at a speed of  $800 \pm 10$  r.p.m., whereby the hose ends fastened to the moving header are rotated at this speed through a circle 8 in. in diameter while the opposite hose ends remain stationary. The end connections on the moveable header are tightly capped, while those on the stationary header are open to a manifold through which water pressure is supplied by means of a weight-operated plunger in a pressure cylinder. The hose assemblies are thereby subjected during test to a constant water pressure which shall be maintained between 225 and 250 psi. as shown by a gage installed so as to read pressure in the manifold. A limit switch operated by the plunger weight must be used to stop the machine when the water pressure drops as in the case of failure of the hose, since it is essential that the machine stop if the pressure drops or a specimen fails. A suitable revolution counter and elapsed time indicator shall be provided.

#### Procedure

14. The nonrotating header shall be equipped to permit attachment of each hose assembly with individual adjustment for length. When mounted in the whip test machine the projected length of the hose assembly shall be  $1.750 \pm 0.015$  in. less than the straight length. The whip test results are very sensitive to errors in setting this length and measurements shall be taken carefully and the reduction from straight length to projected length on the machine shall be within the limits specified. The projected length shall be taken parallel to the axis of the rotating head. The test specimen assemblies shall be installed in the apparatus in their natural "lay," that is, without any twist. The water pressure shall be applied, and all hose and passages bled to eliminate air pockets or bubbles. The motor rotating the moveable head shall then be started and the duration of the test noted. Failure of the specimen by water leakage through a rupture, and consequent loss of pressure, terminates the test. When a minimum time requirement is specified, the machine shall be run continuously during this period.

#### TENSILE STRENGTH TEST

##### Description

15. This tensile strength test consists of subjecting the hose assembly to increasing tension load in a suitable testing machine until failure occurs, either by separation of the specimen from the end fittings or by rupture of the hose structure.

##### Apparatus

16 (a). A tension testing machine conforming to the requirements of the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4) of the American Society for

Testing Materials<sup>4</sup> shall be used for measuring the tensile strength of the hose assembly. The machine shall be provided with a recording device to give the total pull in pounds at the conclusion of the test. A machine of 1000-lb. capacity will be found suitable.

testing machine travels at the rate of approximately 1 in. per min., until the specimen either pulls out of the end fixtures or ruptures. The total load at the time of failure shall be recorded and notation made as to the type of failure.

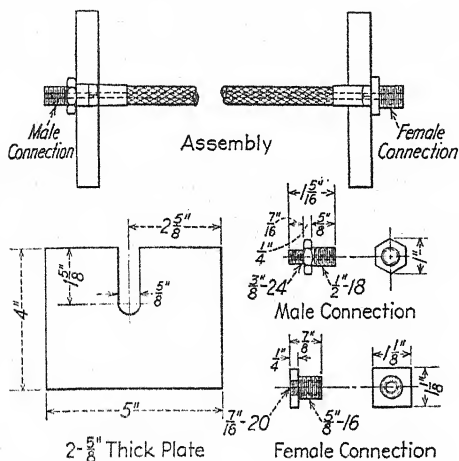


FIG. 4.—Parts to be Assembled on Hose for Tensile Strength Test.

(b) The fixtures for holding the specimens in the testing machine shall be similar to those shown in Fig. 4. The specimen shall be so held that the hose and fittings shall have a straight center-line corresponding to the direction of the machine pull.

#### Procedure

17. The test specimen shall be assembled in the fixtures (Fig. 4) and mounted in the testing machine. A steady tension load shall then be applied, at a speed such that the moving head of the

#### COLD TEST

##### Cold Test

18. The hose shall be conditioned in air in a cold box in a straight position at  $-40^{\circ}\text{F.}$  ( $-40^{\circ}\text{C.}$ ) for 72 hr. After conditioning and without removal from the cold box, the hose shall be bent around a mandrel having a diameter of 3 in. and shall not break or crack.

#### REPORT

##### Report

19. The report shall include the following:

- (1) Identification of the specimen including the free length and type of end fittings,
- (2) Date of test,
- (3) Temperature of test room,
- (4) Calibration correction factors for expansion apparatus,
- (5) The corrected volumetric expansion expressed in hundredths of a cubic centimeter,
- (6) The bursting strength in pounds per square inch,
- (7) The fatigue life, as shown by the whip test, expressed in hours,
- (8) The tensile strength in pounds of total load, and notation of type of failure, and
- (9) Statement of the result of the cold test.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## *Tentative Specifications for*

### FRICION TAPE FOR GENERAL USE FOR ELECTRICAL PURPOSES<sup>1</sup>



A.S.T.M. Designation: D 69 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. These specifications cover friction tape commonly used for protecting and binding in place, insulation applied to joints of electrical wires and cables, and for other electrical and mechanical purposes. The tape consists of cotton sheeting that has been impregnated with an adhesive insulating compound and cut into rolls of narrow width.

#### Cotton Sheeting

2. The cotton sheeting shall be evenly and firmly woven from good cotton and as free from unsightly defects, dirt, knots, lumps, and irregularities of twist as is consistent with the best manufacturing practice. The threads shall run in as straight lines as possible without waving, so as to reduce to a minimum the raveling of the cloth when cut into tape.

#### Frictioning Compound

3. The frictioning compound shall be

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Reverted to tentative by action of the Society at annual meeting, June, 1946.

Prior to their present publication as tentative, these specifications were published as tentative from 1920 to 1924, being revised in 1921 and 1922. They were adopted in 1924, revised in 1927, but withdrawn and republished as tentative from 1932 to 1938, being revised in 1934, 1935, and 1936. They were again adopted in 1938 and published as standard from 1938 to 1945, being revised and republished as tentative in 1946.

a tacky adhesive insulating compound containing practically no free sulfur or other substances which would have a deteriorating effect on copper or other metals, or on the fabric.

#### Impregnation of Fabric

4. The fabric shall be thoroughly impregnated and evenly covered on both sides with the frictioning compound.

#### Adhesiveness of Compound

5. The compound shall adhere firmly to the fabric, and shall not pull away from the fabric so as to leave bare spots when adjacent layers of tape are separated.

#### Tackiness

6. The tape shall show tackiness, that is, ability to stick to itself after light contact has been made, when tested front to back in accordance with the procedure described in Section 21.

#### Discoloration of Copper

7. When the application of the tape is such that the discoloration of the surface to which it is applied is an important consideration, the purchaser may request that the tape to be supplied shall be tested and must pass the test

for discoloration of copper in accordance with the method described in Section 22. At the conclusion of the test the blue-black color, characteristic of copper sulfide, shall not appear on the rod at the edge of the tape.

### Pinholes

8. The number of pinholes in a specimen of three consecutive yards of tape selected at random from any portion of the sample roll shall not exceed the following:

Tape Width, in.	Number of Pinholes, max.
$\frac{3}{4}$ .....	6
1.....	8
$1\frac{1}{2}$ .....	12
2.....	16

### Tensile Strength

9. The tensile strength of the tape specimen shall be not less than 40 lb. per inch of width. The initial distance between the jaws of the testing machine shall be 12 in. and the rate of separation of the jaws shall be 20 in. per min.

### Dielectric Strength

10. Each roll selected for purpose of tests shall be tested for dielectric strength. The breakdown voltage shall be not less than 1000 v.

### Parallelism of the Tape

11. Each roll selected for purpose of tests shall be tested for parallelism of the warp threads with the longitudinal axis in accordance with the procedure described in Section 25. The difference between the compared widths shall not exceed  $\frac{1}{8}$  in.

### Dimensions

12. The tape shall have a nominal thickness of 0.015 in. and shall be made in the following widths:  $\frac{3}{4}$  in., 1 in.,  $1\frac{1}{2}$  in., and 2 in. Each roll shall contain not less than  $82\frac{1}{2}$  ft. and not more than 85 ft. of tape.

### Permissible Variations in Dimensions

13. The thickness shall not vary from that specified by more than plus or minus 0.003 in., nor the width by more than plus or minus  $\frac{1}{32}$  in.

### Packing

14. Each roll of tape shall be enclosed in a paper carton, unless otherwise specified. If enclosed in a carton, either the roll of tape shall be wrapped in a moisture-proof material or the carton shall be wrapped in a moisture-proof material and sealed. The wrapping on the tape or carton shall be secure and shall protect the contents fully.

### Marking

15. Each box shall be marked with the name of the manufacturer or trade-mark, together with the nominal width and length of the tape.

### Inspection

16. The tape shall be inspected and tested either at the place of manufacture prior to shipment or at the place of delivery within 4 weeks from date of delivery.

### Aging and Storage

17. (a) The tape shall at any time within four months after acceptance, if properly stored, meet the cold adhesion test specified in Section 20, except that a weight of 3 lb. per inch of width of tape shall be substituted for the 4 lb. per inch, during unwinding.

(b) Tape, to be properly stored, shall be stored in the original boxes and preferably in a cool, dark location. Tape shall not be stored in close proximity to steam pipes, radiators, or other sources of heat.

### Rejection and Retests

18. (a) The purchaser reserves the right to reject any part of the shipment that does not comply with Sections 14 and 15.

(b) If the tape fails in any one test of those prescribed in these specifica-

tions, two additional rolls shall be selected and subjected to that test. If either of these two additional rolls fails, the lot represented by the test rolls shall be rejected.

(c) The manufacturer shall replace the rejected tape and shall pay freight charges for the return of such material.

#### METHODS OF TESTING

##### Sampling

19. (a) The tape offered for inspection shall be divided into one or more lots of approximately equal numbers of

shall be removed and discarded before taking test specimens.

(d) The tape required for test specimens shall be unwound from the roll at a slow uniform rate without jerking.

##### Cold Adhesion Test

20. (a) The adhesion between adjacent layers of the tape shall be determined as follows: A specimen 23 in. in length shall be removed from the sample roll, care being taken not to touch the surface to be tested with the hands or otherwise. One end of

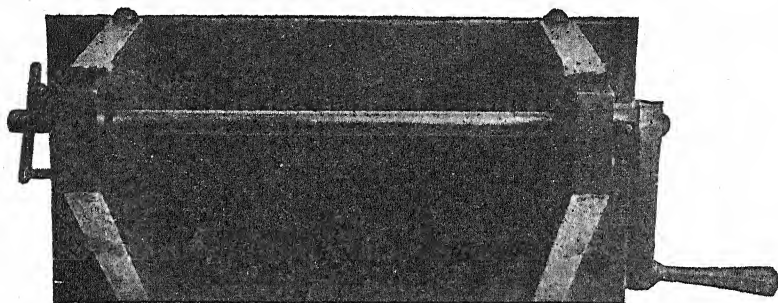


FIG. 1.—Tester for Adhesion Test of Friction Tape.

rolls in accordance with the following requirements:

Rolls Offered for Inspection	Number of Lots
500 and under.....	1
501 to 1500.....	2
1501 to 3000.....	3
Over 3000.....	3 plus 1 for each additional 1500 rolls.

(b) The lots shall be marked for identification and one sample roll shall be taken at random from each lot for purpose of tests and marked to correspond with the lot from which it was selected. Unless otherwise specified, one specimen from each sample roll shall be tested in accordance with the specifications.

(c) At least 2 ft. of the outer layer of each roll selected for purpose of tests

the specimen shall be inserted in the slot of the mandrel described below and 2 in. of the tape shall be wound on the mandrel. A weight of 10 lb. per inch of width shall then be attached to the end of the specimen and 19 in. of the tape shall be wound on the mandrel at a uniform rate of 12 in. per min. The tape shall be allowed to remain 3 min. with the weight attached, after which a weight of 4 lb. per inch of width of tape shall be substituted for the weight of 10 lb. per inch of width, and the tape allowed to unwind. After the first 2 in. have unwound, the length that unwinds in the first 1-min. interval shall be measured and recorded. This length shall not exceed 15 in. The temperature of the room and the temperature of the tape shall be not less than 20 C. (68 F.) nor more than 22.2 C. (72 F.), except

that tape meeting the adhesion test when tested at a higher room temperature shall not be rejected. The relative humidity at the temperature tested shall not exceed 80 per cent.

(b) The mandrel used shall be  $\frac{1}{4}$  in. in diameter with a slot approximately  $\frac{1}{16}$  in. in width and long enough to accommodate the full width of tape, and shall be mounted in a level position in ball bearings of the Fafnir Bearing Co., Catalog No. 36, Serial No. 30, extra small, or equivalent ball bearings. The mandrel shall turn freely under a weight of  $\frac{1}{4}$  oz., suspended from a cotton thread wound in a single layer on the center of the mandrel. See Fig. 1 for a suggested form of tape tester.

#### Tackiness Test

21. (a) New clean specimens shall be taken for all tackiness tests. The temperature of the room and the temperature of the tape and apparatus shall be not less than 20 C. (68 F.) nor more than 22.2 C. (72 F.), except that tape meeting the tackiness test when tested at a higher room temperature shall not be rejected. The relative humidity at the test temperature shall not exceed 80 per cent.

(b) Two 8-in. specimens shall be cut from each sample roll, care being taken not to touch the surface to be tested with the hands or otherwise. One specimen shall be placed lightly on a clean horizontal smooth surface, then the second placed evenly on the first allowing an overlap of  $\frac{1}{2}$  in. lengthwise. Then light pressure shall be applied by the use of a cylindrical glass or chromium-plated brass roller (Note) weighing 9 g.  $\pm$  5 per cent when applied to  $\frac{3}{4}$ -in. tape and proportionately more for wider tape. The roller shall have an outside diameter of not less than  $\frac{1}{2}$  in. and not more than 1 in., and a length to exceed the width of the tape by at least

$\frac{1}{4}$  in. The roller shall be rolled over the specimens once at a slow uniform rate so that the 8-in. length shall be passed over in about 8 sec., care being taken to prevent any downward pressure on the specimens except the weight of the roller.

NOTE.—A suitable apparatus for this test may be made from a squeegee handle and a roller of the proper weight and dimensions. The shaft at the end of the squeegee handle is used to propel the roller by coming in contact with the inside surface of the roller along the horizontal diameter. Small projections or feet at the ends of the shaft and perpendicular to the plane of the handle shall rest on the smooth surface on which the tests are made. This will prevent any downward pressure on the tapes except the weight of the roller.

(c) The two specimens shall then be picked up from the end last in contact with the roller using two pairs of tweezers, one for each specimen. The tackiness shall be such that the point of separation remains approximately in the same horizontal position as the tweezers that pull the specimens apart. In pulling the specimens apart, one hand shall be kept in contact with a rigid support such as the edge of a table and the other hand slowly moved along the edge of the table to bring about the separation of the specimens. The rate at which the specimens are separated shall be steady and shall not exceed 1 in. per second. The last 1 in. of specimen shall be disregarded.

(d) The front to back combination shall be tested three times and if the material fails in more than one test, it shall be considered as having failed the requirement for tackiness.

#### Discoloration of Copper Test

22. The discoloration of copper test shall be made as follows: Five superimposed layers of tape shall be wound on a clean, bright, smooth copper rod, approximately  $\frac{1}{4}$  in. in diameter and 3 in. in length, and baked at 100 C. (212 F.) for 16 hr. in a sealed test tube.

### Determination of Pinholes

23. The number of pinholes shall be determined while the tape specimen is held over a slot in the top of an illuminated box. The box shall be approximately 8 in. in width by 8 in. in height and 18 in. in length, inside dimensions. The slot shall be 2 in. in width by 12 in. in length. The box shall be painted white inside and illuminated by a 25-watt lamp. The slot shall be covered with clear glass set flush with the top of the box. Means shall be provided to limit the light to the width of the tape. Ruptures of the insulating film at the extreme edges of the tape, due to the slight tearing action of the cutting knife, shall not be considered pinholes.

### Dielectric Strength Test

24. The dielectric strength shall be determined by placing a specimen approximately 6 in. in length between two flat electrodes  $\frac{1}{4}$  in. in width by  $4\frac{1}{4}$  in. in length. The edges shall be square and the ends rounded to  $\frac{1}{8}$ -in. radius cylinders whose axes are parallel to the contact faces of the electrode thus giving an area of contact with the surfaces of the tape of exactly  $\frac{1}{4}$  in. in width by 4 in. in length. Under an electrode pressure of 1 psi., a 60-cycle alternating-current voltage of practically sine wave form

shall be applied at a value not exceeding 100 v. and raised at a rate of 100 v. per sec. until puncture occurs. In order to prevent flashover, increased width may be secured by attaching to each side of the test specimen an added piece of tape, making a  $\frac{1}{8}$ -in. lap seam carefully rolled down.

### Parallelism Test

25. The test for parallelism of the tape shall be made as follows: A piece of tape 16 in. in length shall be cut from the sample roll and then torn lengthwise, assisting the tearing by first slitting one end for about  $\frac{1}{2}$  in. with a knife. A 2-in. length shall then be cut from each end of one of the torn halves and the ends of the remaining 12-in. length folded together and compared.

### Measurement of Thickness and Width

26. The thickness of the tape shall be determined by means of a micrometer graduated to 0.001 in. and having a presser foot  $0.25 \pm 0.01$  in. in diameter, exerting a total force of  $9 \pm 0.1$  oz. The load shall be applied by means of a weight. Five thickness and five width measurements shall be made at random in a length of not less than 3 ft. on each sample roll and the maximum, minimum, and average of these five measurements shall be reported.



# *Tentative Specifications for* **RUBBER INSULATING TAPE<sup>1</sup>**



**A.S.T.M. Designation: D 119 - 46 T**

**ISSUED, 1946.<sup>2</sup>**

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## **Scope**

1. These specifications cover rubber insulating tape suitable for the insulation of joints in electrical wires and cables and for other insulating purposes.

## **Compound**

2. The tape shall consist of an unvulcanized or partially vulcanized rubber insulating compound. The compound shall be well, evenly and smoothly calendered, cut to the specified widths and tightly wound in rolls with a separator between layers.

## **Separator**

3. The separator shall be parchment paper or glazed sheeting which shall be interposed between adjacent layers and cover the outside of the tape. The outside end of the roll shall be securely

fastened. Upon originally unwinding the roll the separator shall show no undue tendency to stick to the rubber or to ravel.

## **Composition of Rubber Compound**

4. The rubber compound shall contain not more than 7 lb. of new rubber per 27,000 sq. in. of tape 0.027 in. in thickness.

## **Tensile Strength and Elongation**

5. (a) The tensile strength of the tape shall be not less than 200 psi.

(b) The elongation of a 2-in. gage length determined simultaneously with the tensile strength test shall be not less than 250 per cent.

## **Dielectric Strength**

6. The dielectric strength of the tape shall be not less than 300 v. per mil of thickness.

## **Fusion and Tackiness**

7. The tape shall meet the fusion and tackiness tests specified in Sections 19 and 20

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Reverted to tentative by action of the Society at annual meeting, June, 1946.

Prior to their present publication as tentative, these specifications were published as tentative from 1921 to 1938, being revised in 1922, 1927, 1928, 1932, 1933, 1934, and 1935. They were adopted in 1938 and published as standard from 1938 to 1946. The specifications were revised and republished as tentative in 1946.

**Dimensions**

8. The tape shall conform to the following nominal dimensions:

Width, in.	Thickness, in.	Length, ft.
3	0.027	30
2	0.027	15 <sup>a</sup>
2	0.027	15

<sup>a</sup> Unless otherwise specified on the order, 2-in. tape shall be supplied in rolls having a nominal length of 30 ft.

**Permissible Variations in Dimensions**

9. The thickness shall not vary from the nominal thickness more than plus or minus 0.003 in. and the width not more than plus or minus  $\frac{1}{32}$  in. The length of the tape per roll shall be not less than the nominal length, nor the average length more than 5 per cent in excess of it.

**Packing**

10. Each roll shall be enclosed in a suitable box.

**Marking**

11. Each box shall be marked with the name of the manufacturer or trade mark and the nominal width and length of the tape.

**Inspection**

12. The tape shall be tested and inspected either at the place of manufacture prior to shipment or at the place of delivery within 4 weeks from date of delivery.

**Aging and Storage**

13. (a) The tape shall at any time within four months after acceptance, if properly stored, meet the fusion test specified in Section 19 and shall not become unduly attached to the separator.

(b) Tape, to be properly stored, shall be stored in the original boxes and preferably in a cool, dark location. Tape shall not be stored in close proximity to

steam pipes, radiators, or other sources of heat.

**Rejections and Retests**

14. (a) The purchaser reserves the right to reject any part of the shipment that does not comply with Sections 10 and 11.

(b) If the tape fails in any one test of those prescribed in these specifications, two additional rolls shall be selected and subjected to that test. If either of these two additional rolls fails, the lot represented by the test rolls shall be rejected.

(c) The manufacturer shall replace the rejected tape and shall pay freight charges for the return of such material.

**METHODS OF TESTING****Sampling**

15. (a) All tape offered for inspection shall be divided into one or more lots of approximately equal numbers of rolls in accordance with the following requirements:

Rolls Offered for Inspection	Number of Lots
500 and under.....	1
501 to 1500.....	2
1501 to 3000.....	3
Over 3000.....	3 plus 1 for each additional 1500 rolls

(b) The lots shall be marked for identification and one sample roll shall be taken at random for purpose of tests from each lot and marked to correspond with the lot from which it was selected. Unless otherwise specified, one specimen from each sample roll shall be tested in accordance with the specifications.

(c) At least 2 ft. of tape from each roll shall be removed and discarded before taking test specimens.

**Room Temperature**

16. The temperature of the room in which the tensile strength, elongation and fusion tests are made shall be between 68 and 82 F. (20 and 28 C.).

### Tensile Strength and Elongation Tests

17. The tests for tensile strength and elongation shall be performed in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials,<sup>3</sup> with the following exceptions:

(a) The room temperature shall be as specified in Section 16.

(b) The test specimens shall be cut from a laminated sample, two plies thick, carefully rolled together to avoid

terminated by placing a specimen approximately 6 in. in length between two flat electrodes  $\frac{1}{4}$  in. in width by  $4\frac{1}{4}$  in. in length. The edges shall be square and the ends rounded to  $\frac{1}{8}$ -in. radius cylinders whose axes are parallel to the contact faces of the electrode thus giving an area of contact with the surfaces of the tape of exactly  $\frac{1}{4}$  in. in width by 4 in. in length. Under an electrode pressure of 1 psi., a 60-cycle alternating-current voltage of practically sine wave form shall be applied at a value less than 1000

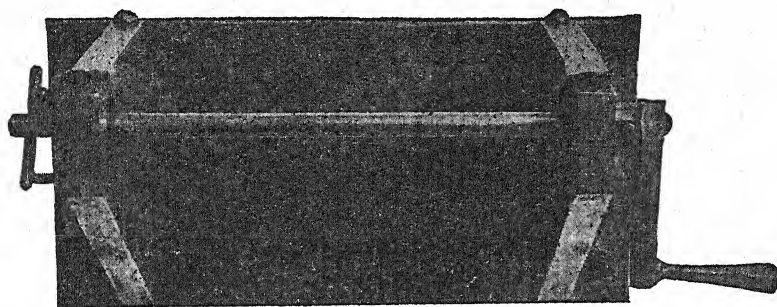


FIG. 1.—Tester for Fusion Test.

entrapped air. The thickness of the composite specimen shall be used in calculating the tensile strength.

(c) The dumbbell-shaped  $\frac{1}{2}$  by 2-in. test specimen shall be used and shall be cut with the A.S.T.M. standard die as shown in Figs. 2 and 3 (Die A) of Standard Methods D 412, except that the ends of the specimen cut from  $\frac{3}{4}$ -in. tape need not be full width. In placing the parallel gage marks on the test specimen care shall be taken not to cut the stock.

(d) Three specimens, free from visible defects, from each sample roll shall be tested. The average of the three tests shall be reported.

### Dielectric Strength Test

18. The dielectric strength shall be de-

v. and raised at the rate of 1000 v. per sec. until puncture occurs. In order to prevent flashover, increased width may be secured by attaching to each side of the test specimen an added piece of tape, making a  $\frac{1}{8}$ -in. lap seam carefully rolled down.

### Fusion Test

19. (a) A specimen 7 in. in length shall be taken from the roll, care being observed not to touch the surface to be tested with the hands or otherwise. Two inches of the specimen shall be wound on the mandrel, and then a weight of 4 lb. per inch of width of tape shall be attached to the end of the specimen. Should this weight be insufficient to elongate the tape 200 per cent or more, additional downward force

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

shall be applied by hand until 200 per cent elongation has been produced. The tape shall then be wound on the mandrel at a uniform rate of 12 in. per min. and allowed to stand for 3 min. with the weight attached. Then, with the weight still attached, the tape shall be allowed to unwind slowly. The mandrel shall revolve not more than two and one-half turns in the first 2 min., during which time the tape shall sustain the weight or shall break at the point of unwinding.

(b) The mandrel used shall be  $\frac{1}{4}$  in. in diameter and shall be mounted in a level position in ball bearings of the Fafnir Bearing Co. Catalog No. 36, Serial No. 30, extra small or equivalent ball bearings. The mandrel shall turn freely under a weight of  $\frac{1}{4}$  oz., suspended from a cotton thread wound in a single layer on the center of the mandrel. See Fig. 1 for a suggested form of tape tester.

#### Tackiness Test

20. A specimen 7 in. in length shall be taken and wound on the mandrel in the same manner as described in Section 19. Instead of leaving the weight suspended for 3 min. it shall be immediately removed. The tape shall show no tendency to unwind from the mandrel.

#### Measurement of Thickness and Width

21. The thickness of the tape shall be determined by means of a micrometer graduated to 0.001 in. and having a presser foot  $0.25 \pm 0.01$  in. in diameter, exerting a total force of  $9 \pm 0.1$  oz. The load shall be applied by means of a weight. Five thickness and five width measurements shall be made at random in a length of not less than 3 ft. on each sample roll and the maximum, minimum and average of these five measurements shall be reported.

# *Tentative Methods of*

## TESTING RUBBER-COATED FABRICS<sup>1</sup>



A.S.T.M. Designation: D 751 - 46 T

ISSUED, 1943; REVISED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods describe procedures for the testing of rubber-coated fabrics made by applying either natural rubber compounds or those synthetic elastomers which are processed on rubber machinery.

### CONDITION

#### Standard Condition

2. (a) *Test Condition*.—Coated fabrics shall be tested in standard condition, unless otherwise specified.

(b) *Standard Condition*.—Standard condition of coated fabrics shall be that reached by the fabric when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A tolerance of plus or minus 2 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

(c) *Moisture Equilibrium*.—It shall be considered that moisture equilibrium is reached when, after free exposure to air in motion, there is no progressive increase in weight. Moisture equilib-

rium shall be approached from the dry side (not moisture free). Certain coatings may greatly retard moisture penetration into the fabric and, therefore, fabrics coated on both sides may require 24 hr. or more to reach equilibrium.

### DIMENSIONS AND WEIGHT<sup>3</sup>

#### Length

3. (a) *General Method*.—The coated fabric shall be laid out smooth, without tension, on a horizontal surface and the length shall then be measured parallel to the selvage; or, successive portions, each at least 5 yd. in length shall be measured under the same conditions.

(b) *Drum Method*.—The coated fabric shall be run over a measuring drum with just enough uniform tension to keep it running flat and true. The length shall be determined from the dial or counter on the drum and its calibration.

#### Width

4. The width shall be measured with

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Administrative Committee on Standards, September 9, 1946.

<sup>3</sup> The procedures for dimensions and weight, breaking strength, elongation, and tearing strength by the trapezoid method are identical with those appearing in the Standard General Methods of Testing Woven Textile Fabrics (A.S.T.M. Designation: D 39), 1946 Book of A.S.T.M. Standards, Part III-A.

the coated fabric laid out smooth on a horizontal surface but without tension in either direction. The average of at least five different measurements uniformly distributed along the full length of a roll or piece shall be reported as the average width of the roll or piece.

### Thickness

5. (a) *Thickness Gage.*<sup>4</sup>—The gage used for the measurement of thickness shall be of the deadweight type equipped with a dial graduated to read directly to 0.001 in. The presser foot shall be circular with a diameter of  $0.375 \pm 0.001$  in. The presser foot and moving parts connected therewith shall be weighted so as to apply a total load of  $6 \pm 0.1$  oz. (equivalent to a pressure of 3.4 psi.) to the specimen. The presser foot and anvil surfaces shall be plane to within 0.0001 in. and parallel to each other to within 0.0001 in. The gage shall be calibrated for the actual load exerted by the presser foot by means of any device so arranged as to measure the total vertical force exerted by the presser foot at the several gage readings or presser foot levels selected for calibration. The presser foot shall be brought to each calibration level from a higher one.

NOTE.—It is suggested that the calibration may be accomplished by supporting the presser foot on a horizontal strap suspended from a weighing device so arranged that it may be raised or lowered gradually and leveled at any required position.

(b) *Procedure.*—The coated fabric shall be placed upon the anvil of the gage smooth, but without tension. The presser foot shall be lowered upon the material gradually (without impact) allowed to rest upon it for 10 sec.,

and the reading of the dial then observed. Similar measurements shall be made at not less than five different places uniformly distributed over the surface of the coated fabric exclusive of the area adjacent to either selvage and within one-tenth the width of the fabric or within 3 yd. of either end of a roll or piece. The average of the five or more measurements shall be reported as the average thickness.

### Weight

6. (a) *Method Applicable to a Piece, Cut, or Roll.*—The full piece, cut, or roll shall be weighed on a calibrated scale, the length and width of the coated fabric shall be measured, and the weight per linear yard or per square yard calculated.

(b) *Method Applicable to a Sample.*—A specimen having an area of at least 20 sq. in. (or a number of specimens not less than 2-in. square and having a total area of at least 20 sq. in.) shall be cut from the coated fabric, weighed on a calibrated scale, and the weight per linear yard or per square yard shall be calculated. Unless a specimen the full width of the fabric is used, no specimen shall be taken nearer the selvage than one-tenth the width of the fabric.

NOTE.—This method is intended for use when a small sample of coated fabric is sent to the laboratory for test. The result is considered to be applicable to the sample, but not to the piece or lot of goods from which the sample was taken unless the number of samples and method of sampling are specified and agreed upon by those concerned. If this is done, each sample should be tested in accordance with Paragraph (b) and the results averaged to obtain the average weight per square yard or per linear yard of the lot.

### BREAKING STRENGTH<sup>3</sup>

#### Breaking Strength

7. Breaking strength shall be determined by the Grab Method, unless the Strip Method is specified. In the case of coated knit fabrics, the breaking

<sup>4</sup> The requirements for the thickness gage, pendulum type testing machine and fabric clamps for the breaking strength test, and the bursting strength testers are identical with those appearing in the Standard Specifications for Textile Testing Machines (A.S.T.M. Designation: D 76), 1946 Book of A.S.T.M. Standards, Part III-A.

strength may be more satisfactorily determined by the bursting strength method which shall be used when specified.

#### A. Grab Method

##### Testing Machine<sup>4</sup>

8. (a) The testing machine shall be of the pendulum type in which the specimen is strained by a uniform movement of the pulling clamp to which one end of the specimen is attached, the other end being held in a clamp attached to a weighing device based on the principle of the pendulum.

(b) The machine shall be power-driven, or shall be operated in such a manner that the pulling clamp shall have a speed of  $12 \pm \frac{1}{2}$  in. per min., unless otherwise specified. A machine shall be considered to be of proper capacity for a test, if, when the specimen breaks, the angle which the pendulum makes with the vertical is between 9 and 45 deg. The minimum pitch diameter of drum for transferring the pull on the specimen to the pendulum shall be  $2\frac{1}{2}$  in.

(c) Vertical machines of this type shall be calibrated throughout the loading range of a 9-to 45-deg. swing of the pendulum from the vertical position using a series of weights of known amount applied in an ascending order. The weights shall be fastened to the upper clamp in any convenient manner and suspended freely, thereby causing the pendulum to swing from the vertical position. The equilibrium position of the pendulum for each weight shall be determined by disengaging the pawls, bringing the pendulum slightly below its equilibrium position, engaging the pawls, and then releasing the pendulum and allowing it to swing up to the equilibrium position with the pawls engaged. The average of several readings for each weight shall be used to determine the dial and chart errors.

##### Clamps for Fabrics<sup>4</sup>

9. The clamps for holding a specimen of coated fabric shall have metallic gripping surfaces sufficiently smooth, flat, and parallel as to prevent the test specimen from slipping or moving between the gripping surfaces when held under the pressure normal to operation. The dimension of all gripping surfaces parallel to the direction of application of the load shall be 1 in.; the dimension perpendicular to this direction shall be 1 in. for the face jaw and 2 in. or more for the other. All edges which might cause a cutting action shall be rounded to a radius of not over  $\frac{1}{8}$  in. The design of the clamp shall be such that one gripping surface shall be an integral part of the rigid frame of the clamp while the other shall be on a part hinged or swiveled to the movable member of the clamp. The pressure between the gripping surfaces, sufficient to clamp the specimen firmly before the testing load is applied and to prevent slippage during the progress of the test, shall be secured by any suitably constructed mechanical device operating on the movable member of the clamp. The distance between the clamps at start of test shall be 3 in.

NOTE.—As a practical method of determining the degree of flatness and parallelism of the assembled clamp mechanism it is recommended that a sheet of thin white paper, between two thin sheets of carbon paper, be placed between the gripping surfaces, and the jaws then brought together with a light pressure.

##### Test Specimens

10. Specimens 4 in. in width and not less than 6 in. in length shall be cut from the coated fabric for test. Two sets of five specimens each will be required, one set for longitudinal breaking strength having the longer dimension parallel to the lengthwise direction of the goods, and the other set for transverse breaking strength, having the longer dimension



parallel to the crosswise direction of the goods. No two specimens shall contain the same threads of the fabric in the direction to be tested. Unless otherwise specified, specimens shall be taken no nearer the selvage than one-tenth the width of the coated fabric.

#### Procedure

11. The specimen shall be placed symmetrically in the clamps of the machine (see Fig. 1) with the longer dimension parallel to and the shorter dimension at right angles to the direction of application of the load. The average of the results of the five individual tests in each direction shall be reported as the

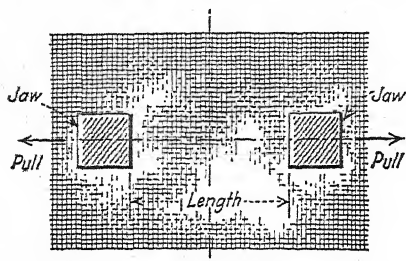


FIG. 1.—Illustration of Grab Test.

longitudinal breaking strength and the transverse breaking strength of the fabric respectively. If a specimen slips in the clamps, breaks in the clamps, breaks at the edge of the clamps, or if for any reason attributable to faulty operation, the result falls markedly below the average for the set of specimens, the result shall be discarded, another specimen taken, and the result of the break included in the average.

#### B. Cut Strip Method

##### Procedure

12. The breaking strength test by the cut strip method shall be made in accordance with the directions for the grab method (Sections 8 to 11), with the exception that the specimens shall be cut 1 in. in width (or other width when specified).

#### ELONGATION<sup>3</sup>

##### Procedure

13. Unless otherwise specified, the elongation of a coated fabric at any stated load shall be obtained when the breaking strength (Sections 7 to 12) is determined and for the same specimens by means of a suitable autographic recording device on the testing machine. The elongation shall be the average of the results obtained for five specimens, and it shall be expressed as the percentage increase in length. Since the initial length and, therefore, the measured elongation depend upon the load applied in placing the specimen in the clamps of the machine, an initial load of 6 oz., or other initial load specified for the particular material in question, shall be placed on the specimen before gripping the specimen in the lower clamp of the machine. The elongation shall be calculated from the start of the line as shown on the graphic record.

#### BURSTING STRENGTH

##### Testing Machine<sup>4</sup>

14. The machine used for determining bursting strength shall be either of the following types.

(a) *Pendulum Machine with Ring Clamp*.—The pendulum machine described in Section 8 shall be equipped with a bursting attachment of such design that the specimen held securely by a ring-clamp mechanism  $1.750 \pm 0.001$  in. in internal diameter with the center of the specimen pressed against a polished steel ball of  $1.000 \pm 0.001$  in. in diameter until a burst is produced. The direction of motion of the ring clamp shall be at right angles to the initial plane of the specimen. The capacity of the machine shall be considered suitable if, when the specimen breaks, the angle which the pendulum makes with the vertical is between 9 and 45 deg.

(b) *Diaphragm Bursting Tester*.—The diaphragm bursting tester shall conform to the following requirements:

(1) The testing machine to be used may be either mechanically or manually operated. It shall permit the clamping of the material to be tested between two circular clamps not less than 3 in. in diameter having coaxial apertures of their centers  $1.200 \pm 0.001$  in. in diameter. The surfaces of the clamps between which the specimen is to be placed shall be eccentrically grooved. The grooves shall be spaced not less than  $\frac{1}{32}$  in. apart and of a depth not less than 0.006 in. The grooves shall not start closer than 0.125 in. from the edge of the aperture. The surfaces of the clamps shall be metallic and any edge which might cause a cutting action shall be rounded to a radius of not over  $\frac{1}{16}$  in. The lower clamp shall be integral with the chamber in which a screw shall operate to force a liquid pressure medium at a uniform rate of  $3.00 \pm 0.25$  cu. in. per min. against a rubber diaphragm fitted to expand through the aperture exerting its force against the coated fabric set between the two clamps. (In the manually operated machine, this shall correspond to approximately 60 rpm. of a hand wheel turning the displacement screw.)

(2) In the mechanically operated machine, means shall be provided for stopping, at the instant of rupture of the specimen, any further application of the loading pressure, and for holding unchanged the contents of the pressure chamber until the gross bursting pressure and tare diaphragm pressure, simultaneously indicated on the gage have been recorded. The machine shall be fitted with a Bourdon tube gage maximum hand type, having a dial 4.5 in. in diameter, the scale divided to read in 0.5-lb. units, with a

range of from 0 to 100 lb. Accuracy shall be within plus or minus 0.25 lb. When the gage is calibrated, it shall be mounted in the same relative position as on the bursting tester. If calibrated with a dead-weight tester, the weights shall be spun slowly to insure free action of the piston.

(3) Provision shall be made for applying to the specimen, before clamping, an initial tension which shall be uniform in all directions.

### Test Specimens

15. At least ten test specimens shall be cut, the smallest dimension of which shall be at least 0.5 in. greater than the outside diameter of the ring-clamp mechanism of the testing machine.

### Procedure

16. (a) When the pendulum machine is used, the test specimen shall be mounted in the ring clamp, which shall then be moved at a rate of 12 in. per min. against the steel ball until rupture occurs. The pounds pressure shall be recorded as the bursting strength of the specimen.

(b) When the diaphragm tester is used, the specimen shall be mounted between the ring clamp and the base surface with a uniform tension. The pressure shall be applied at a constant rate as prescribed in Section 14 (b) Item (1) until rupture occurs. The pounds pressure shall be recorded as the bursting strength of the specimen.

NOTE.—When the diaphragm bursting tester is used, it is recommended that the difference between the gross bursting pressure and the tare diaphragm pressure, be reported as the bursting strength of the material.

### Calculation

17. The average bursting strength of the coated fabric shall be the arithmetic mean of the results of ten tests.

## TEARING STRENGTH

## Tearing Strength

18. The tearing strength of the coated fabric shall be determined by the trapezoid method except that in the case of light-weight materials, the pendulum impulse method may be used when specified.

*A. Trapezoid Method<sup>3</sup>*

## Testing Machine

19. The testing machine used for this test shall conform to the requirements of the machine used in the grab method for breaking strength with the following modifications: The faces of the clamps shall measure 1 by 3 in. (Section 8) or more, with the longer dimension perpendicular to the direction of application of the load. The distance between the clamps at the start of the test shall be 1 in. The pawls on the pendulum shall be disengaged from the ratchet.

## Test Specimens

20. The test specimens shall be 3 in. in width and 6 in. in length. Two sets of five specimens will be required, one set for longitudinal tearing strength, having the longer dimension parallel to the lengthwise direction of the coated fabric and the other set for transverse tearing strength, having the longer dimension parallel to the crosswise direction of the coated fabric. An isosceles trapezoid having an altitude of 3 in. and bases 1 and 4 in. in length shall be marked on each specimen preferably with the aid of a template. A cut  $\frac{1}{4}$  to  $\frac{3}{8}$  in. in length shall then be made in the center of the 1-in. edge and perpendicular to it.

## Procedure

21. The specimen shall be clamped in the machine along the nonparallel sides of the trapezoid so that the cut is halfway between the clamps and the

short edge shall be held taut, the long edge lying in folds. The machine shall be started and the average load necessary to tear the coated fabric shall be observed, preferably by means of an autographic recording device. The average of the results of the five individual tests in each direction shall be reported as the longitudinal tearing strength and the transverse tearing strength of the fabric, respectively.

*B. Pendulum Impulse Method<sup>5</sup>*

## Apparatus

22. (a) The testing machine shall be of the pendulum impulse type and shall consist of the following:

(1) A stationary clamp,

(2) A movable clamp carried on a pendulum, preferably formed by a sector of a wheel or circle, free to swing on a ball bearing or other substantially frictionless bearing,

(3) Means for holding the pendulum in a raised position and means for releasing it instantaneously, and

(4) Means for registering the maximum arc through which the pendulum swings when so released. The pendulum shall carry a circumferential scale, graduated from 0 to 100 so as to read against the pointer the work done in gram-centimeters by the pendulum when tearing a specimen, divided by 137.6 (one-sixteenth of the force in grams required to tear a specimen).

(b) With the pendulum in its initial position ready for a test, the two clamps shall be separated by an interval of 2.5 mm. (0.10 in.) and so aligned that the specimen clamped in them lies in a plane perpendicular to the plane of oscillation of the pendulum with the edges of the jaws gripping the coated fabric in a horizontal line, a perpendicular to which

<sup>5</sup> The pendulum impulse method for tearing strength is substantially identical with the Standard Method of Test for Internal Tearing Resistance of Paper (A.S.T.M. Designation: D 689), see p. 436.

through the axis of suspension of the pendulum is 104 mm. (4 in.) in length and makes an angle of 27.5 deg. with the plane of the coated fabric.

(c) The clamping surface in each jaw shall be over 25 mm. (1 in.) in width and over 12 mm. (0.5 in.) in depth.

#### Adjustment of Apparatus

23. (a) For apparatus having a sector as a pendulum, and which tears the specimen as it moves toward the right, draw a pencil line on the base or stop-mechanism 1 in. to the right of the edge of the sector stop. With the sector raised to its initial position and the pointer set against its stop, on releasing the sector and holding the stop down, the sector should make at least 20 complete oscillations before the edge of the sector which engages with the stop no longer passes to the left of the pencil line. Otherwise, the bearing shall be oiled and adjusted.

(b) Level the instrument so that, with the sector free, the line on the sector indicating the vertical from the point of suspension coincides with a corresponding point on the base of the instrument, usually placed on the stop mechanism. After leveling, operate the instrument several times with nothing in the jaws, the movable jaw being closed, to ascertain if the pointer registers zero with no load. If zero is not registered, the pointer stop should be suitably adjusted until the zero reading is obtained. If it is necessary to move the pointer stop, the pointer friction should be checked as follows: Set the pointer at the zero reading on the scale before releasing the sector and after release see that the pointer is not pushed more than three scale divisions beyond the zero. A reading of more than three divisions indicates excessive pointer friction and the pointer should be removed, the bearing wiped clean, and a trace of oil or petroleum jelly applied. When the pointer friction

has been reduced, finally adjust the point stop.

#### Calibration of Apparatus

24. (a) Level and adjust the instrument on a level sheet of plate glass and clamp a known weight in grams,  $W$ , to the radial edge of the sector beneath the jaws. The center of gravity of the weight (including means of attaching) shall be previously marked by a punched dot on the face of the weight that is to be in the front of the instrument.

(b) Raise and set the sector as for tearing a specimen and, by means of a surface gage or other convenient means, measure the height in centimeters,  $H$ , of the center of gravity of the weight above the glass plate. Then release the sector, allow it to swing, and note the pointer reading. Without touching the pointer, raise the sector until the edge of the pointer just meets with its stop, in which position again determine the height in centimeters,  $h$ , of the center of gravity of the weight above the glass plate.

(c) The work done is  $W(H - h)$  gram-centimeters. The pointer reading for the standard instrument and method specified should be as follows:

$$\frac{W(H - h)}{137.6}$$

(d) Five weights from 75 to 400 g form a suitable range for the calibration, one or more being clamped on the edge of the sector in different positions, the work done in raising each being calculated and added together.

(e) A record shall be made of deviations of the indicated readings and corresponding corrections made in the test results.

(f) It is unnecessary to repeat the calibration of the instrument provided it is kept in adjustment and no parts become changed or worn, but the tearing

distance, which equals 43 mm. (1.69 in.) shall be checked prior to each series of tests and adjusted if necessary.

### Test Specimens

25. Test specimens shall be cut accurately in each principal direction of the coated fabric about 76 mm. (3 in.) in length by exactly 63 mm. (2.5 in.) in width with the slit to be cut 20 mm. (0.8 in.) in length, leaving exactly 43 mm. (1.69 in.) between the end of the slit and the edge of the specimen.

### Procedure

26. (a) The testing machine shall be levelled and adjusted, if necessary, before each set of tests.

(b) The test specimen shall be placed midway in the clamps with its upper edge parallel to the top of the jaws and so that the initial slit is at right angles to the top of the jaws. When torn, the scale readings should be between 20 and 90. If over 90, an auxiliary weight of 2501 g. shall be attached to the pendulum which doubles the capacity of the machine. Not less than five tests in each principal direction of the coated fabric shall be made.

(c) If the mean value of the lowest and the highest reading differs from the average of all the readings by more than 10 per cent, additional specimens shall be tested until there is agreement within these limits. An isolated very high or low result that is not repeated in duplicate when a consistent average has been obtained without the abnormal reading shall be discarded. If the tear deviates more than 10 mm. ( $\frac{3}{8}$  in.) from the line of the initial slit, a notation should be made and the deviation reported. If the side of the specimen above the movable clamp rubs against the sector as a tear is made, that reading shall be rejected.

### Calculation and Report

27. (a) The results shall be reported as the force in pounds required to tear a

specimen. Since the scale readings are made 28.6 times as great as the actual values, the tearing resistance values shall be calculated by dividing the average instrument reading (corrected if necessary for calibration error) by 28.6. When the auxiliary weight is used, the scale reading shall be divided by 14.3. If the results are desired to be expressed in grams required to tear a specimen, the scale reading shall be multiplied by 16 and if the auxiliary weight is used, multiplied by 32.

(b) The average, maximum, and minimum results for both principal directions of the coated fabric shall be reported. These shall be designated as longitudinal tear resistance and transverse tear resistance, respectively.

### HYDROSTATIC RESISTANCE

#### Hydrostatic Resistance

28. The hydrostatic resistance of a coated fabric shall be determined by Method A which uses a modified diaphragm bursting tester (Mullen test) or, when specified, by Method B which uses the hydrostatic pressure of a rising column of water (Suter test).

#### *Method A. Pressure Application by Modified Diaphragm Bursting Tester*

#### Testing Machine

29. (a) The hydrostatic resistance of a coated fabric shall be determined on a diaphragm bursting tester as described in Section 14 (b), except that the diaphragm shall be removed, allowing the liquid used for displacing the diaphragm to come in direct contact with the specimen. The liquid shall be water.

(b) An interchangeable upper clamp having a glass plate fitted in its aperture shall be provided for use when specified or as described in Paragraph (c). The glass shall be not less than 0.125 in. in thickness. The upper clamp shall be of the tripod type to assure maximum

visibility of the material to be tested. A lever type of clamping mechanism with a screw and spring assembly may be provided to permit rapid and proper clamping of the material to be tested.

(c) In cases where the bursting strength of the coated fabric is less than 30 psi. or in the case of knit fabrics where the "give" of the fabric might exceed the elastic limit of the coating, or where other factors might make it desirable, a screen of 16 mesh per square inch shall be placed directly over the specimen and used in conjunction with the glass-fitted upper clamp. For easy detection of the leak, a filter paper may be placed over the screen prior to clamping.

#### Test Specimens

30. The test specimen cut from the coated fabric shall be of such size that the smallest dimension is at least 0.5 in. greater than the outside diameter of the ring-clamp mechanism of the testing machine.

#### Procedure

31. Before clamping the specimen into the testing machine, the water level shall be brought up flush with the surface of the circular opening in the tester so that no air pocket may exist between the water surface and the coated fabric being tested. Unless otherwise specified in the case of materials coated on one side only, the coated side shall be placed next to the water level. In the case of double-coated fabrics or double-texture fabrics, either surface may be used unless the side to be put facing downward against the water level is definitely specified. The temperature of the water shall be the same as the atmospheric temperature of the testing room. The test shall be conducted in accordance with one of the following two procedures, except that when the screen and glass support is employed only Procedure 2 shall be used.

*Procedure 1.*—The pressure shall be increased steadily at the rate of speed indicated in Section 14 (b) and a dial reading shall be taken at the first appearance of water through the coated fabric being tested. Ten different determinations shall be made and the average taken as the hydrostatic resistance of the coated fabric. An isolated high or low result that is not repeated in duplicate when a consistent average has been obtained without the abnormal reading shall be discarded and a retest substituted.

*Procedure 2.*—A specified pressure held constant within plus or minus 2.0 lb. shall be applied against the test specimen for a period of 5 min. Any appearance of water through the coated fabric that can be detected during that period will indicate failure to meet the test. At least ten specimens from any sample of material shall be tested. The coated fabric will have satisfactorily met the required test if at least eight out of ten tests show no leakage at the pressure specified.

NOTE.—Any material which fails to give a value of at least 5 lb. as measured by either procedure of testing shall be regarded as having too low a hydrostatic resistance to be measured by this method and the alternate Method B of using the hydrostatic pressure of a rising column of water shall be tried.

#### Report

32. The report shall include the following:

(1) Statement of the method and procedure used and whether or not the screen and glass support was employed.

(2) With Procedure 1, all observed and recorded data together with the average value of the hydrostatic resistance in pounds per square inch.

(3) With Procedure 2, statement of the number of specimens tested, the specified pressure applied, and the number of specimens which withstood the pressure.



*Method B. Pressure Application by a Rising Column of Water*

**Testing Machine**

33. (a) The machine shall consist of a clamp for holding the specimen and a means of providing a column of water whose level can be raised. Figure 2 shows a suitable apparatus.

(b) The clamp shall consist of a lower ring  $6\frac{3}{4}$  in. in outside diameter by  $4\frac{1}{2}$  in. in inside diameter held in a fixed position and provided with a channel  $\frac{3}{8}$  in. in width into which a rubber gasket seats, and also a movable upper plate which shall be  $6\frac{3}{4}$  in. in outside diameter and shall carry the rubber gasket. A water chamber on top of the upper plate shall have a  $\frac{1}{2}$  in. water inlet, a  $\frac{3}{4}$  in. water-opening into the clamp, and a  $\frac{1}{4}$  in. vent pipe. A suitable mechanism shall be provided to close the clamp with the specimen of fabric between the ring and plate.

(c) The water leveler shall be provided with a water inlet, water outlet, and overflow pipe. It shall be connected to the clamp by means of a rubber hose  $\frac{1}{2}$  in. in inside diameter. The overflow pipe provides a means of maintaining a constant level of water. A suitable mechanism for raising the water leveler uniformly at the required rate and lowering it again shall be provided. A chain and sprocket device driven by a motor and controlled by a suitable disk clutch has been found satisfactory.

(d) A steel scale for measuring the height of the water column shall be attached to the water leveler in such a way that the zero is in the same horizontal plane as the constant water level at all times. A reference pointer in fixed position at the level of the face of the lower ring of the clamp shall be provided for measuring the distance of travel of the water leveler above the face of the clamp. The scale shall be graduated in both centimeters and inches and shall be

capable of being read to the nearest millimeter or sixteenth of an inch.

(e) A mirror shall be provided under the clamp in order to observe any leaks in the fabric being tested.

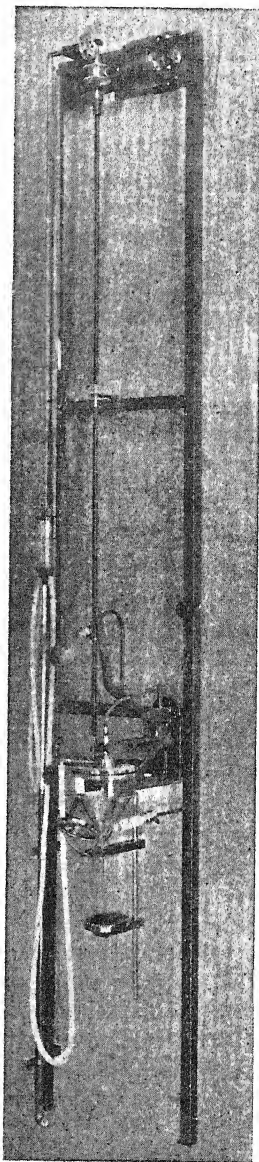


FIG. 2.—Machine for Measuring Hydrostatic Resistance of a Coated Fabric by Using the Pressure of a Rising Column of Water.



### Test Specimens

34. The test specimen of the coated fabric shall be at least 8 in. square. The specimen may include a seam if desired since the rubber gasket provides a means of obtaining a tight seal in the clamp. At least five specimens from any sample of material shall be tested.

### Procedure

35. The test shall be conducted according to one of the following two procedures:

*Procedure 1.*—The specimen shall be laid smoothly on the face of the lower ring of the clamp, and the upper plate lowered and fastened in place. This shall form a water-tight compartment. When the specimen is coated on only one side, the coated side shall be placed next to the water column, unless otherwise specified. With the leveler at the zero position, the water shall be turned on and kept running at such a rate as to overflow continuously. The column shall then be raised at a rate of 1 cm. per sec. until the first drop of water appears through the coated fabric. The height of the water column above the level of the specimen shall then be read on the scale.

*Procedure 2.*—An alternative procedure may be used by placing the specimen in the clamp and raising the water column to a predetermined height and recording the time required for the first drop of water to penetrate the coated fabric.

### Report

36. The report shall include the following:

(1) Statement of the method and procedure used and the number of specimens tested,

(2) With Procedure 1, the height of the column of water in inches at which the first drop appeared on the under

side of each of the coated fabric specimens, also the average value for all specimens,

(3) With Procedure 2, the preset height of the column of water and the time elapsed before the first drop of water appeared on the under side of each specimen of the coated fabric, also the average value for all specimens.

### ADHESION OF COATING TO FABRIC

#### Testing Machine

37. A pendulum type testing machine as described in Section 8 (b) shall be used except that the speed of the pulling clamp shall be 2 in. per min. A machine shall be considered to be of proper capacity for the test, if, when the coating separates from the fabric, the angle which the pendulum makes with the vertical is between 9 and 45 deg.

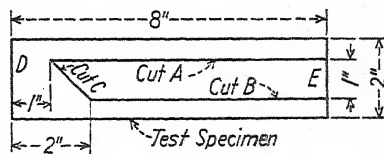


FIG. 3.—Test Specimen Showing Cuts for Coating Adhesion Test.

#### Test Specimens

38. Specimens 2 in. in width and 8 in. in length shall be cut from the coated fabric. Two sets of two specimens each will be required, one set for adhesion in the longitudinal direction having the longer dimension parallel to the lengthwise direction of the fabric, and the other set for adhesion in the transverse direction having the longer dimension parallel to the crosswise direction of the fabric. These specimens shall be prepared for test as follows:

(a) When the strength of the coating film exceeds the adhesive bond to the fabric, as with thick films, the specimen

shall be prepared for test by carefully cutting the coating through to the fabric on two parallel cuts *A* and *B*, 1 in. apart running lengthwise of the specimen as shown in Fig. 3. Cut *A* shall extend from the end of the specimen, *E*, to within 1 in. of the opposite end, *D*. Cut *B* shall extend from end *E* to within 2 in. of end *D*. The ends of cuts *A* and *B* shall be joined together with a diagonal cut *C* which shall also be carefully cut through the coating to the fabric. The edge of a knife shall be worked under the point formed by cuts *A* and *C* and the coating of the 1 in. strip separated from the backing for a distance of 2 in. from that point.

(b) With thin films or in cases where the coating is not sufficiently strong to be stripped from the fabric as described in Paragraph (a), two specimens of the coated fabric shall be cemented together face to face with an adhesive suitable for adhering the type of coating being evaluated. The cuts shown in Fig. 3 shall then be made from one side being careful to penetrate only through one layer of fabric and not to injure the other fabric layer. The fabric from one side shall then be stripped down for a distance of 2 in. This will allow the backing fabric to be inserted in one jaw of the machine, and a double thickness of coating and a backing fabric from the other piece to be inserted in the other jaw of the machine.

The separation between the coating and the backing fabric can then be measured as noted in Paragraph (a).

NOTE.—In case the fabric is too weak to be tested in a 1-in. width, the specimen may be cut 3 in. in width and the strip for evaluation cut 2 in. in width. The results shall then be recorded as pounds pull per 2-in. strip.

### Procedure

39. The separated portion of the 1-in. strip shall be clamped in the lower jaw of the testing machine and end *D* of the specimen clamped in the upper jaw so that the movement of the lower jaw will separate the coating from the backing. The rate of travel of the lower jaw shall be 2 in. per min. The pawls shall be disengaged and the pendulum allowed to swing freely. The average pull in pounds of the two specimens cut in the longitudinal direction shall be reported as the adhesion of the coating in the longitudinal direction, and the average pull in pounds of the other two specimens as the adhesion of the coating in the transverse direction of the fabric.

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NOTE.—When a test for hydrogen permeability is required, reference should be made to the Tentative Method of Test for Hydrogen Permeability of Rubber-Coated Fabrics (A.S.T.M. Designation: D 815).<sup>6</sup>

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<sup>6</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# *Tentative Method of Test for*

## HYDROGEN PERMEABILITY OF RUBBER-COATED FABRICS<sup>1</sup>



A.S.T.M. Designation: D 815 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is intended for use in the determination of the permeability of hydrogen into air through fabrics coated with rubber or synthetic elastomers.

### Apparatus

2. The apparatus required is the Cambridge fabric permeameter or its equivalent. The essential components, properly assembled for the test as shown in Fig. 1, shall comprise the following:

(a) *Waxing Plate Assembly*.—A waxing plate assembly as shown in Fig. 2, consisting of two metal plates 5 in. in diameter mounted in a holder so that the fabric specimen may be clamped between them with a peripheral ring  $\frac{5}{16}$  in. in width exposed for wax coating.

(b) *Test Plates*.—Test plates essentially in two parts, the lower half being the hydrogen chamber and the upper half containing the analysis cell. The specimen to be tested shall be sandwiched

between the upper and lower plates. The analysis cell is part of a Wheatstone bridge arrangement, which is contained in the control box. The hydrogen diffusing into the analysis cell produces an "unbalance" in the system which is registered by the galvanometer.

(c) *Control Box*.—A control box, essentially a Wheatstone bridge, with a standard cell. A 6-v. storage battery or any other means of supplying the proper voltage with steady current to the operating circuit may be used.

(d) *Galvanometer*.—A galvanometer of a sensitive type and calibrated by the manufacturer to read in liters of hydrogen per square meter per 24 hr. at a stated temperature.

### Test Temperature

3. The test shall be conducted at the temperature at which the apparatus is calibrated within a tolerance of plus or minus 5 F. All specimens, equipment, and gases shall be preconditioned at this temperature for 1 hr. prior to test. Unless otherwise specified, 77 F. shall be taken as the standard temperature for calibration and test. A thermometer

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, December 13, 1944.

shall be provided at the back of the lower test plate for measurement of the actual temperature while testing.

### Test Specimens

4. The test specimens shall be accurately cut from the coated fabric in circular form,  $5\frac{5}{8}$  in. in diameter. This may be done conveniently by means of a die having the correct dimensions. Three specimens from each sample shall

to have failed to conform to the specifications.

### Preparation of Specimens

5. The waxing compound shall be prepared by melting together a mixture of half beeswax and half pure petrolatum in a small tank. Each specimen shall be centered carefully between the waxing plates and clamped tightly by means of the threaded clamp plug. The ex-

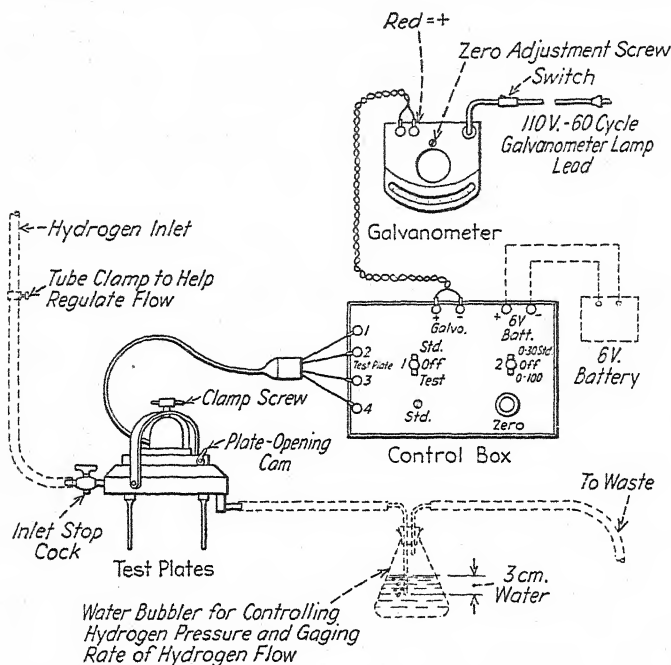


FIG. 1.—Fabric Permeameter Showing General Arrangement of Set-Up for Testing with Air as Reference.

be tested, and the average of the readings taken as the permeability except when a minimum requirement is established, as in testing for compliance with a particular specification, in which case only one test shall be required. If the result of this test is below the specified requirement, two additional specimens shall be prepared from the original sample and tested. Should the results of either of these tests be below the specified requirement, the sample shall be considered

posed edge of the specimen shall then be rotated in the melted waxing compound until it is coated with a thin uniform layer of the wax. Care shall be taken that the cut edge is well sealed by the wax. As soon as the wax is sufficiently hard, the waxing plates shall be opened and the specimen removed. Waxing serves three purposes: (a) to define the test area exactly, (b) to give a good seal between plates, and (c) to prevent the

leakage of hydrogen through the edge of the fabric.

### Balancing Period

6. The apparatus is designed to give direct readings of permeability after a preliminary balancing period, which has the effect of bringing the operation onto the straight line portion of a curve of permeability plotted against time. Characteristic curves shall be made for each grade of material to determine the length of the balancing period. For this purpose, the

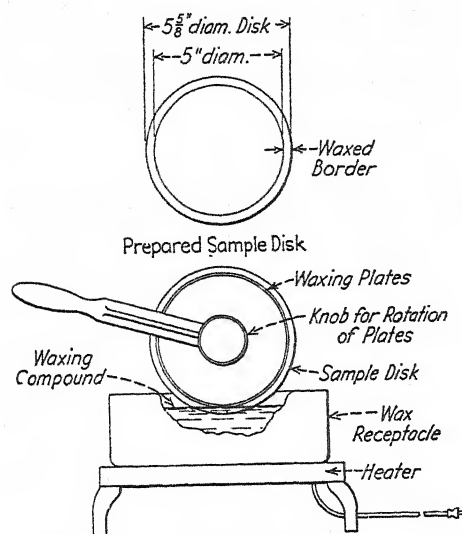


FIG. 2.—Preparation of Samples for Testing.

assembled apparatus shall be first adjusted in accordance with the manufacturer's instructions so that the galvanometer reads correctly at zero and at maximum deflection. A specimen of the material prepared for test shall then be clamped between the test plates with moderate pressure. Care shall be taken to see that the edges of the plates are free of accumulated wax and that the plate-opening cam is raised so that it does not hold the plates apart (Note). The galvanometer shall then be brought to zero by means of the zero adjustment,

after which the hydrogen valve shall be opened and the rate of flow set at approximately 200 ml. per min. or more for 1 min. to sweep out the air from the lower chamber. The time of opening of the hydrogen valve shall be noted and taken as the start of the balancing period. The flow of hydrogen may be determined with sufficient accuracy by noting the rate of bubbling through a water bottle. A pressure of .3 cm. of water shall be maintained on the specimen by immersing the end of the hydrogen outlet tube 3 cm. below the surface in the water bottle. After the first minute the rate of flow shall be cut down to approximately 50 ml. per min. This rate will assure the maintenance of 100 per cent hydrogen on the lower side of the specimen. Starting at the end of the sweeping out period, galvanometer readings of permeability shall be taken at intervals of 30 sec. and plotted against time on graph paper until the resulting graph is a straight line. The length of time after the initial starting of the hydrogen flow until the curve becomes a straight line plus 1 min. shall be taken as the proper balancing period to assure that subsequent tests will be conducted on the straight line portion of the curve.

NOTE.—As the wax on the edges of the test specimens tends to build up between the test plates after several specimens have been tested, it is necessary to remove it at frequent intervals with a suitable solvent such as carbon tetrachloride, using care to avoid introduction of solvent into the analysis cell. This will prevent errors due to change of volume in the test chamber or leakage of hydrogen caused by uneven accumulations. After using the solvent, however, it is necessary to ventilate the upper test plate sufficiently so that any solvent or vapors which may have entered the analysis cell are replaced with air.

### Procedure

7. In carrying out a test, the specimen shall be clamped between the test plates and the hydrogen flow started as in the

determination of the balancing period (Section 6). The time shall be noted and the flow continued for the duration of the balancing period. Since this is not critical, it need not be timed closer than within a tolerance of a few seconds. The galvanometer should be kept approximately on the zero but need not be balanced exactly to zero during this period. As the end of the balancing period approaches, the galvanometer spot shall be brought slightly to the left of the zero mark and allowed to approach it without further adjustment. The exact time the spot crosses the zero line shall be taken as the start of the actual test using a stop watch or other suitable timing device. The galvanometer shall then be allowed to deflect for exactly 2 min. without further balancing and at the end of the 2-min. period the permeability shall be read directly from the galvanometer scale. Since the instrument scale is calibrated for a 2-min. reading, this interval must not be varied unless the scale reading is multiplied by a suitable correction factor (Note). After completion of the test, the hydrogen flow shall be stopped and all switches thrown to the "off" position, after which

the specimen shall be removed from the test plates. Before inserting the next specimen for test, the upper plate shall be left swung back on its hinge for at least a minute in order to allow air to replace all the hydrogen in the analysis cell. After inserting the next specimen, it is not necessary to rebalance the instrument, provided the balancing period is expected to be the same. The spot will be off scale at the beginning of each run, but this is immaterial, provided it is made to approach zero in time for the beginning of the next 2-min. reading.

NOTE.—It may be found that some materials of high permeability exhibit a characteristic curve which begins to droop so early that the straight line portion lasts less than 2 min. In such cases a 1-min. reading should be taken and multiplied by two.

#### Report

8. The report shall include the following:

- (1) Description of the material tested, including thickness and type of coating,
- (2) Temperature of test, and
- (3) Permeability expressed in liters of hydrogen per square meter per 24 hr..

*Tentative Specifications for*  
**INSULATED WIRE AND CABLE: CLASS AO, 30 PER CENT  
HEVEA RUBBER COMPOUND<sup>1</sup>**



**A.S.T.M. Designation: D 27 - 46 T**

ISSUED, 1946; REVISED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Scope**

1. These specifications cover the conductors, insulation, cable tape, cotton braid, and lead sheaths of Class AO rubber insulated wire and cable. The design and factor of safety depend upon the service conditions and shall be at the option of the purchaser.

**CONDUCTOR**

**Material**

2. The conductor shall be soft annealed copper properly coated with tin, lead, or lead-alloy, and shall have the properties and characteristics specified in Sections 3 to 7. Coatings conducive to galvanic action at splices, such as zinc, are not recommended.

**Strand**

3. Each individual wire of a stranded

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Reverted to tentative by action of the Society at the Annual Meeting, June, 1946; revision accepted by the Administrative Committee on Standards, September 9, 1946.

Prior to their present publication as tentative, these specifications were published as tentative from 1916 to 1941, being revised in 1916, 1921, 1928, 1931, 1933, 1934, 1935, 1936, 1937, 1938, 1939, 1940, and 1941. They were adopted in 1941, published as standard from 1941 to 1946, when they were revised and republished as tentative.

conductor shall be considered separately and shall be designated as a strand.

**Shape**

4. Each solid conductor and each strand shall be round and reasonably free from imperfections.

**Stranding**

5. The stranding shall conform to the Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (A.S.T.M. Designation: B 8) Table II, class B or, where specified, class C.<sup>3</sup> If a more flexible stranding is specified in the order, it shall conform to the requirements prescribed in the Tentative Specifications for Rope-Lay-Stranded Copper Conductors Having Bunch-Stranded Members for Electrical Conductors (A.S.T.M. Designation: B 172),<sup>3</sup> or for Rope-Lay-Stranded Copper Conductors Having Concentric-Stranded Members for Electrical Conductors (A.S.T.M. Designation: B 173),<sup>3</sup> or for Bunch-Stranded Copper Conductors for Electrical Conductors (A.S.T.M. Designation: B 174).<sup>3</sup>

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part I-B.



### Permissible Variations in Diameter and Area

6. When the diameter of solid conductors and strands is specified, the permissible variation from the specified value shall not exceed 1 per cent under or 3 per cent over for wire 0.010 in. in diameter or larger, and 0.1 mil under or 0.3 mil over for wire less than 0.010 in. in diameter. When the area of cross-section of stranded conductors is specified, the permissible variation shall not exceed 1 per cent under the specified value.

### Tinning Test

7. (a) Where lead or lead-alloy coated wire is used the continuity of the lead coating shall be determined in accordance with Section 7 of the Tentative Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (A.S.T.M. Designation: B 189).<sup>3</sup>

(b) *Retests and Rejections.*—If the specimens tested in accordance with Paragraph (a) show any signs of failure, two more specimens shall be tested. If one of these two additional specimens fail, that coil or reel shall be rejected. If both specimens pass the test for continuity of coating, the coil or reel shall be accepted. If more than 10 per cent of the samples in the entire order fail, all of the wire shall be rejected. If 10 per cent or less of the samples in the entire lot fail, each coil, reel, or length may be tested and accepted or rejected upon the results of the individual tests.

## INSULATION

### Methods of Test

8. (a) *Chemical Analysis.*—The analytical tests necessary for determining conformity to the chemical composition requirements in Section 11 shall be made in accordance with the complete procedure as described in the Tentative

Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297) of the American Society for Testing Materials.<sup>4</sup>

(b) *Physical and Electrical Tests.*—All measurements and tests necessary for determining conformity of the insulation to these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470)<sup>4</sup> except as otherwise prescribed in these specifications.

### Rubber Compound

9. (a) Wires and cables ordered under these specifications shall be insulated with Class AO rubber compound.

(b) Where performance rubber compound is desired, the order shall be placed under the Standard Specifications for Insulated Wire and Cable: Performance Rubber Compound (A.S.T.M. Designation: D 353),<sup>4</sup> where heat-resisting compound is desired, under the Standard Specifications for Insulated Wire and Cable: Heat-Resisting Rubber Compound (A.S.T.M. Designation: D 469),<sup>4</sup> and where ozone-resistant type insulation is desired, under the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (A.S.T.M. Designation: D 574)<sup>4</sup> of the American Society for Testing Materials.

### Composition of Compound

10. (a) Class AO rubber compound shall contain exclusively not less than 30 per cent nor more than 33 per cent of the best quality Hevea rubber which has not previously been used in a rubber compound, solid waxy hydrocarbons, suitable mineral matter, antioxidants, organic accelerators, and sufficient sulfur to properly vulcanize the compound.

(b) The mineral matter shall be dry.

<sup>4</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

(c) The waxy hydrocarbons used shall be solid at 54 C. and shall be free from saponifiable matter.

(d) Contamination of the compound, such as by the use of impregnated tapes, will not excuse the manufacturer from conforming to these specifications.

### Chemical Requirements

11. (a) *Quantitative Requirements.*—The insulation shall consist of a properly vulcanized rubber compound which after vulcanization shall conform on analysis to the following requirements expressed as percentages by weight of the whole sample:

Rubber, per cent. ....	30 to 33
Waxy hydrocarbons, max., per cent. ....	4
Free sulfur, max., per cent. ....	0.7

	30 per cent Rubber <sup>a</sup>	33 per cent Rubber <sup>a</sup>
Saponifiable acetone extract, max., per cent. ....	2.00	2.20
Unsaponifiable resins, max., per cent. ....	1.00	1.10
Chloroform extract, max., per cent. ....	0.90	1.00
Alcoholic potash extract, max., per cent. ....	0.55	0.60
Total sulfur, max., per cent. ....	2.10	2.30
Specific gravity, min. ....	1.75 <sup>a</sup>	1.67 <sup>a</sup>

<sup>a</sup> For percentages between 30 and 33, the specific gravity limits shall be in proportion to the percentage of rubber found.

(b) *Qualitative Requirements.*—The acetone solution shall not fluoresce. The hydrocarbons shall be solid, waxy, and not darker than a light brown. The chloroform extract (60 ml.) shall not be darker than a straw color.

### Sampling for Analysis

12. Samples of the vulcanized rubber compound for chemical analysis shall be taken after the successful completion of the physical and electrical tests, and before applying impregnating compounds which may contaminate the rubber. The samples shall be taken at the option of the purchaser. The

purchaser shall assure himself that all samples are free from contamination and change due to torch heating. Wherever practicable, samples shall be taken at a distance of at least 3 ft. from the end of the wire.

### Physical Properties

13. The insulation shall conform to the requirements as to physical properties prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min., psi. ....	1200
Tensile stress at 200 per cent elongation, min., psi. ....	300
Elongation at rupture, min., per cent. ....	400
Set in 2-in. gage length, max., in. ....	$\frac{3}{4}$
Tensile strength and elongation after 96 hr. in air oven test at 157 to 159 F. (69.4 to 70.6 C.), min., per cent of original value ....	85
Tensile strength and elongation after 96 hr. in oxygen pressure test, min., per cent of original value ....	75

### High Voltage Test

14. Each coil, reel, or length of wire or cable, after vulcanization shall successfully withstand the application of an alternating-current voltage of not less than that prescribed in Table II for a period of 5 min.

### Insulation Resistance

15. (a) Each coil, reel, or length of wire or cable after being subjected to the high voltage test specified in Section 14 shall have an insulation resistance not less than that prescribed in Table III.

(b) In calculating insulation resistance, the temperature coefficients given in Table IV shall be used.

### Moisture Absorption

16. (a) The increase in the specific inductive capacity of the insulation shall be used as an indication of the amount of moisture absorbed.

(b) The specific inductive capacity after 24-hr. immersion in water shall not exceed six. The increase between the ends of the first and the fourteenth days shall not exceed 20 per cent and that

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For non-leaded submarine cables,  $\frac{1}{2}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits except where one side of a delta system is permanently grounded, in which case, multiply the circuit voltage (phase to phase) by 1.73 and use the resulting voltage to select the corresponding insulation thickness.

NOTE 5.—Above 5000 v. alternating-current, ozone-resistant insulation is recommended as prescribed in the Tentative Specifications for Insulated Wire and Cable: Ozone-Resistant Type Insulation (A.S.T.M. Designation: D 574) of the American Society for Testing Materials.<sup>a</sup>

Rated Circuit Voltage	Size of Conductor, A.w.g. Numbers or Circular Mils	Insulation Thick- ness on Each Conductor, sixty- fourths of an inch	Test Voltage
0 to 300 .....	No. 18 to No. 16 .....	2	1 000
	No. 14 <sup>a</sup> to No. 12 <sup>a</sup> .....	2	1 500
	No. 16 to No. 9 <sup>b</sup> .....	3	3 000
	No. 8 to No. 2 .....	4	3 500
0 to 600 .....	No. 1 to No. 0000 .....	5	4 000
	225 000 to 500 000 cir. mils. ....	6	5 000
	525 000 to 1 000 000 cir. mils. ....	7	6 000
	Over 1 000 000 cir. mils. ....	8	7 000
	No. 14 to No. 8 .....	4	5 000
	No. 7 to No. 2 .....	5	6 000
601 to 1000 .....	No. 1 to No. 0000 .....	6	7 500
	225 000 to 500 000 cir. mils. ....	7	9 000
	525 000 to 1 000 000 cir. mils. ....	8	10 000
	Over 1 000 000 cir. mils. ....	9	11 000
	No. 14 to No. 8 .....	5	6 000
	No. 7 to No. 2 .....	6	7 500
1001 to 2000 .....	No. 1 to No. 0000 .....	7	9 000
	225 000 to 500 000 cir. mils. ....	8	10 000
	525 000 to 1 000 000 cir. mils. ....	9	11 000
	Over 1 000 000 cir. mils. ....	9	11 000
	No. 10 to No. 8 .....	7	9 000
	No. 7 to No. 2 .....	8	10 000
2001 to 3000 .....	No. 1 to No. 0000 .....	8	10 000
	225 000 to 500 000 cir. mils. ....	9	11 000
	525 000 to 1 000 000 cir. mils. ....	9	11 000
	Over 1 000 000 cir. mils. ....	10	12 500
	No. 10 to No. 8 .....	9	11 000
	No. 7 to No. 2 .....	9	11 000
3001 to 4000 .....	No. 1 to No. 0000 .....	9	11 000
	225 000 to 500 000 cir. mils. ....	10	12 500
	525 000 to 1 000 000 cir. mils. ....	10	12 500
	Over 1 000 000 cir. mils. ....	11	13 500
	No. 8 .....	10	12 500
	No. 7 to No. 2 .....	10	12 500
4001 to 5000 .....	No. 1 to No. 0000 .....	10	12 500
	225 000 to 500 000 cir. mils. ....	11	13 500
	525 000 to 1 000 000 cir. mils. ....	11	13 500
	Over 1 000 000 cir. mils. ....	12	15 000
	No. 8 to No. 0000 .....	10	12 500
5001 to 6000 .....	225 000 to 1 000 000 cir. mils. ....	11	13 500
(grounded) .....	Over 1 000 000 cir. mils. ....	12	15 000
	No. 8 to No. 0000 .....	12	15 000
5001 to 6000 .....	225 000 to 1 000 000 cir. mils. ....	12	15 000
(ungrounded) .....	Over 1 000 000 cir. mils. ....	13	16 500
	No. 8 to No. 0000 .....	11	13 500
6001 to 7000 .....	225 000 to 1 000 000 cir. mils. ....	11	13 500
(grounded) .....	Over 1 000 000 cir. mils. ....	12	15 000
	No. 8 to No. 0000 .....	14	17 500
6001 to 7000 .....	225 000 to 1 000 000 cir. mils. ....	14	17 500
(ungrounded) .....	Over 1 000 000 cir. mils. ....	15	19 000
	No. 8 to No. 0000 .....	12	15 000
7001 to 8000 .....	225 000 to 1 000 000 cir. mils. ....	12	15 000
(grounded) .....	Over 1 000 000 cir. mils. ....	13	16 500
	No. 8 to No. 0000 .....	16	20 000
7001 to 8000 .....	225 000 to 1 000 000 cir. mils. ....	16	20 000
(ungrounded) .....	Over 1 000 000 cir. mils. ....	17	21 000

<sup>a</sup> Building Wire with  $\frac{3}{16}$ -in. wall is permitted in sizes 14 and 12 by the National Electrical Code and will be furnished unless otherwise specified.<sup>b</sup> Sizes Nos. 16 and 18 are not recognized by the National Electrical Code for 600 v. service.

between the ends of the seventh and fourteenth days shall not exceed 5 per cent.

(c) The specific inductive capacity

measured at 60 cycles, the voltage impressed upon the conductor shall be equivalent to a stress of between 30 to 40 v. per mil of insulation.

TABLE III.—INSULATION RESISTANCE.

Insulation Thickness In Sixty-fourths of an Inch.....			2	3	4	5	6	7	8	9	10	12
Size of Conductor, A.w.g. numbers or circular mils	Solid or Stranded	Diameter, in.	Insulation Resistance, megohms—1000 ft. at 60 F. (15.5 C.)									
No. 18.....	Solid	0.040	8600	11 100	.....	.....	.....	.....	.....	.....	.....	.....
No. 16.....	Solid	0.051	7300	9 500	.....	.....	.....	.....	.....	.....	.....	.....
No. 14.....	Solid	0.064	.....	8 300	9900	11 300	12 550	13 650	14 600	15 450	16 250	17 650
No. 12.....	Solid	0.081	.....	7 050	8550	9 850	11 000	12 000	12 900	13 750	14 500	15 850
No. 10.....	Solid	0.102	.....	6 000	7350	8 500	9 600	10 500	11 350	12 100	12 850	14 150
No. 9.....	Solid	0.111	5490	6 770	7900	8 900	9 810	10 630	11 380	12 080	12 730	13 330
No. 8.....	Solid	0.128	.....	.....	6200	7 300	8 300	9 150	9 950	10 650	11 350	12 550
No. 7.....	Solid	0.144	.....	5 720	6730	7 640	8 460	9 220	9 920	10 570	11 180	11 750
No. 6.....	Stranded	0.184	.....	.....	4750	5 600	6 400	7 200	7 900	8 500	9 100	10 200
No. 4.....	Stranded	0.232	.....	.....	3900	4 700	5 450	6 100	6 700	7 250	7 800	8 850
No. 2.....	Stranded	0.292	.....	.....	3250	3 900	4 550	5 150	5 650	6 150	6 650	7 600
No. 1.....	Stranded	0.332	.....	.....	.....	3 550	4 100	4 650	5 150	5 600	6 100	6 950
No. 0.....	Stranded	0.373	.....	.....	.....	3 200	3 750	4 200	4 700	5 150	5 550	6 350
No. 00.....	Stranded	0.418	.....	.....	.....	2 900	3 400	3 850	4 300	4 700	5 100	5 850
No. 000.....	Stranded	0.470	.....	.....	.....	2 600	3 100	3 500	3 900	4 250	4 650	5 400
No. 0000.....	Stranded	0.528	.....	.....	.....	2 400	2 800	3 150	3 550	3 900	4 200	4 900
250 000 cir. mils.....	Stranded	0.575	.....	.....	.....	.....	2 600	2 950	3 300	3 650	4 000	4 600
350 000 cir. mils.....	Stranded	0.681	.....	.....	.....	.....	2 250	2 550	2 850	3 150	3 450	4 000
500 000 cir. mils.....	Stranded	0.814	.....	.....	.....	.....	1 900	2 200	2 450	2 700	2 950	3 450
750 000 cir. mils.....	Stranded	0.998	.....	.....	.....	.....	.....	1 800	2 050	2 250	2 500	2 900
1 000 000 cir. mils.....	Stranded	1.152	.....	.....	.....	.....	.....	1 600	1 800	2 000	2 200	2 600
1 250 000 cir. mils.....	Stranded	1.289	.....	.....	.....	.....	.....	.....	1 600	1 800	2 000	2 350
1 500 000 cir. mils.....	Stranded	1.412	.....	.....	.....	.....	.....	.....	1 500	1 650	1 850	2 150
1 750 000 cir. mils.....	Stranded	1.526	.....	.....	.....	.....	.....	.....	1 400	1 550	1 700	2 000
2 000 000 cir. mils.....	Stranded	1.631	.....	.....	.....	.....	.....	.....	1 300	1 500	1 600	1 900

Insulation Thickness in Sixty-fourths of an Inch.....

13

14

15

16

17

Size of Conductor, A.w.g. numbers or circular mils	Solid or Stranded	Diameter, in.	Insulation Resistance, megohms—1000 ft. at 60 F. (15.5 C.)				
No. 9.....	Solid	0.114	13 900	14 430	14 940	15 420	15 870
No. 8.....	Solid	0.128	13 100	13 600	14 100	14 600	14 900
No. 7.....	Solid	0.144	12 280	12 790	13 270	13 720	14 160
No. 6.....	Stranded	0.184	10 650	11 150	11 500	12 000	12 450
No. 4.....	Stranded	0.232	9 300	9 700	10 100	10 500	10 900
No. 2.....	Stranded	0.292	7 950	8 400	8 800	9 150	9 500
No. 1.....	Stranded	0.332	7 350	7 650	8 000	8 400	8 750
No. 0.....	Stranded	0.373	6 750	7 050	7 400	7 800	8 100
No. 00.....	Stranded	0.418	6 200	6 500	6 900	7 150	7 500
No. 000.....	Stranded	0.470	5 700	6 150	6 350	6 650	6 900
No. 0000.....	Stranded	0.528	5 200	5 500	5 800	6 050	6 350
250 000 cir. mils.....	Stranded	0.575	4 900	5 200	5 450	5 750	5 950
350 000 cir. mils.....	Stranded	0.681	4 250	4 500	4 700	4 950	5 200
500 000 cir. mils.....	Stranded	0.814	3 700	3 950	4 150	4 350	4 550
750 000 cir. mils.....	Stranded	0.998	3 100	3 300	3 500	3 700	3 900
1 000 000 cir. mils.....	Stranded	1.152	2 750	2 950	3 100	3 300	3 450
1 250 000 cir. mils.....	Stranded	1.289	2 500	2 650	2 850	3 000	3 150
1 500 000 cir. mils.....	Stranded	1.412	2 300	2 450	2 600	2 700	2 950
1 750 000 cir. mils.....	Stranded	1.526	2 150	2 300	2 450	2 600	2 750
2 000 000 cir. mils.....	Stranded	1.631	2 050	2 200	2 300	2 450	2 600

NOTE 1.—This table is based on a constant,  $K = 21,120$  in the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where  $R$  = the insulation resistance in megohms—1000 ft.,

$K$  = constant,

$D$  = diameter over insulation, on each conductor, and

$d$  = diameter over conductor.

NOTE 2.—For an intermediate size, the insulation resistance shall be that of the next larger size.

shall be determined at either 1000 or 60 cycles. When measured at 1000 cycles, the voltage impressed upon the conductor shall not exceed 10 v. When

Thickness

17. (a) The average thickness of the insulation shall be not less than that

prescribed in Table II. The minimum thickness shall be not less than 95 per cent of the thickness required by Table II for insulations less than  $\frac{1}{8}$  in. in thickness and not less than 90 per cent of the specified thickness for insulations of  $\frac{1}{8}$  in. and greater.

(b) The thicknesses prescribed in Table II apply to single-conductor cable and the individual conductors of multiple-conductor cables, leaded or braided, except special applications such as aerial, nonleaded submarine, and vertical riser cable.

TABLE IV.—TEMPERATURE COEFFICIENTS.

Temperature			Temperature		
Fahren- heit Deg.	Centi- grade Deg.	Tem- pera- ture Coeffi- cient	Fahren- heit Deg.	Centi- grade Deg.	Tem- pera- ture Coeffi- cient
46	7.8...	0.69	61	16.1...	1.03
47	8.3...	0.71	62	16.7...	1.05
48	8.9...	0.73	63	17.2...	1.08
49	9.4...	0.75	64	17.8...	1.11
50	10.0...	0.77	65	18.3...	1.14
51	10.6...	0.79	66	18.9...	1.17
52	11.1...	0.81	67	19.4...	1.20
53	11.7...	0.83	68	20.0...	1.23
54	12.2...	0.85	69	20.6...	1.26
55	12.8...	0.88	70	21.1...	1.30
56	13.3...	0.90	71	21.7...	1.33
57	13.9...	0.92	72	22.2...	1.37
58	14.4...	0.94	73	22.8...	1.40
59	15.0...	0.97	74	23.3...	1.44
60	15.6...	1.00	75	23.9...	1.48

NOTE.—In Appendix II will be found an interpretation of the insulation thicknesses given in Table II.

(c) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one

determination of the thickness made on each coil or reel so selected.

(d) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any one point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement shall be made after slicing off the heavier side of the insulation.) In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

### Workmanship

18. (a) The insulation shall be homogeneous in character, tough, elastic, and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(b) *Shielding.*—Wires and cables not supported on insulators shall be shielded or suitably protected against corona in accordance with the detailed recommendations contained in Sections 20 to 24, inclusive.

(c) *Covering.*—The insulation of braided wires up to No. 7 A.w.g., inclusive, shall be covered with a single cotton braid. In multiple-conductor cables the individual conductors shall not be braid-covered unless so specified. On larger sizes the fibrous covering shall consist of a cable tape and a cotton braid or two cotton braids. The cable tape

shall conform to the requirements specified in Sections 25 to 27, inclusive. The cotton braid shall be applied in accordance with Sections 28 to 34, inclusive, and shall be finished in accordance with Sections 34 to 45.

(d) *Repairs or Joints*.—Where repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 17.

### Rejection

19. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

(b) The purchaser may make a chemical analysis on any one of the samples selected as described in Section 12 to determine if the compound conforms to the requirements of Section 11. Failure of any sample selected to conform to the requirements shall be sufficient cause for rejecting the wire which that sample represents, except in the case of failure to conform to the requirements for either or both free sulfur and chloroform extract. In case of such failure, the coil from which the sample was taken shall be rejected and two additional samples shall be selected from the remainder of the order. The remainder of the order shall be accepted if both samples so selected conform to the requirements for free sulfur or chloroform extract. The entire order shall be rejected if either sample fails.

(c) When ten or more samples are selected in any inspection lot, all coils, reels, or lengths may be rejected if more

than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. When the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the result of such individual tests.

(d) Each coil, reel, or length which fails to conform to with the electrical requirements prescribed in these specifications shall be rejected.

### SHIELDING

#### Types

20. When shielding is used, metallic shields are most suitable. In some cases, when safety to life is not involved, local conditions permit the use of the semi-conducting type of shield, as in power station and substation wiring.

#### Application

21. When shielding is specified for multiple conductor cables, the shield shall be applied over the individual conductors, except when shielding is required for reasons of safety only, in which case the shield may be applied over the conductor assembly.

#### Metallic Shield

22. The metallic shield should be a nonmagnetic tape of suitable width and at least 0.0025 in. in thickness. It shall be applied so as to substantially cover the surface of the insulation. If applied in an open spiral, the maximum space between successive turns shall not exceed 10 per cent of the tape width. The edges of the tape, where in contact with the insulation, shall be substantially free from burrs, and joints



shall be made electrically continuous by welding, soldering, or by suitable overlapping. The shielding tape shall be applied directly over the insulation or fibrous tape or tapes. Metallic tape shall be suitably protected against chemical action from contact with the insulation.

### Recommended Shielding Practice

23. Shielding is recommended at three-phase operating voltages (line to line) above those prescribed in Paragraphs (a) and (b) and Section 24, except as otherwise specified.

(a) *Metallic Covered Cable.*—Multiple conductor cables having lead sheath or other metallic coverings shall be shielded for operation at three-phase potentials (line to line) above 10 kv. grounded neutral and 6 kv. ungrounded neutral. For submarine cables shielding may be used throughout but shall be used on shore ends.

(b) *Nonmetallic Covered Cable.*—Single conductor and multiple conductor cables having nonmetallic coverings shall be shielded for operation at three-phase potentials (line to line) above 6 kv. grounded neutral and 3.5 kv. ungrounded neutral. This shall not apply to cables installed on insulators or for special conditions.

### Special Conditions for Shielding

24. (a) Single and two conductor cables installed in ducts or metal conduits, unless operating under permanently dry conditions, shall be shielded at all operating potentials above 2 kv. whether grounded or ungrounded neutral.

(b) Aerial cables installed in metal rings shall be shielded at all operating potentials above 2 kv. whether grounded or ungrounded neutral.

(c) Aerial cables installed in marlin ties shall be shielded for operation at three-phase potentials (line to line)

above 6 kv. grounded neutral and 3.5 kv. ungrounded neutral.

(d) Conditions under which shielding is necessary for protection to cables connected to overhead lines, or otherwise subjected to induced potentials and where protection from accidental shocks is necessary, shall be treated as individual special cases.

NOTE.—For single-phase and two-phase operation the equivalent voltage shall be determined in accordance with Appendix II.

### CABLE TAPE

#### Material

25. Cable tape made from cotton cloth of the following constructions may be used:

	Weight, lb. per sq. yd.	Number of Yards per Pound (40-in. Width)	Thread Count
Alternate (a) . . . . .	0.225	4.00	60 by 52
Alternate (b) . . . . .	0.210	4.30	56 by 48

The tape shall be treated on one side with an insulating compound of a nature not injurious to the wire insulation. The minimum thickness shall be 0.010 in. Other types of tape may be used if approved by the purchaser.

#### Application

26. The tape shall be applied helically and may be applied before vulcanization. The maximum width and minimum overlap shall conform to the values in Table V.

TABLE V.—WIDTH AND OVERLAP OF RUBBER-FILLED CABLE TAPE.

Diameter Over Insulation, in.	Maximum Width of Tape, in.	Minimum Overlap, in.	Minimum Overlap, per cent
0 to 0.250 . . . . .	1½	$\frac{3}{16}$	12.5
0.251 to 0.500 . . . . .	2	$\frac{1}{4}$	12.5
0.501 to 0.750 . . . . .	2½	$\frac{5}{16}$	12.5
0.751 to 1.00 . . . . .	3	$\frac{3}{8}$	12.5
1.001 to 1.500 . . . . .	4	$\frac{1}{2}$	12.5
1.501 to 2.000 . . . . .	5	$\frac{5}{8}$	12.5

#### Number of Tests

27. Samples shall be taken at the option of the purchaser.



## COTTON BRAID

## Types of Braids

28. Three types of cotton braid are covered in these specifications, as follows:

*Code Braid*, designed for indoor service and installation in conduits.

*Standard Braid*, designed for indoor service and installation in conduits.

*Heavy Braid*, designed for outdoor and rough service.

## Code Braid

29. Unless otherwise specified, code braid shall be supplied.

## Material

30. Cotton braids shall be closely woven from cotton yarn. Standard braid shall be woven from cotton yarn having not less than two plies. Single ply yarn may be used for code braid if the diameter under the braid is 0.8 in. or less. For diameters greater than 0.8 in. and for heavy braids, yarn of at least two plies shall be used.

## Construction

31. (a) The braid shall be so constructed that the tangent of the angle between the cotton thread and the side of the wire shall be not less than that specified in Table VI.

TABLE VI.—TANGENT OF ANGLE OF BRAID.

Diameter Under Braid, in.	Value of Tangent	Corresponding Braid Angle, deg.
0 to 1.00.....	0.700	35
1.001 to 1.500.....	0.839	40
1.501 and over.....	1.000	45

(b) The thickness of code braid shall be not less than that specified in Table VII. The thickness of standard braid shall be not less than that specified in Table VIII and that of heavy braid not less than that specified in Table IX.

TABLE VII.—MINIMUM THICKNESS OF CODE BRAID.

Diameter Under Braid, in.	Corresponding Minimum Size and Ply of Cotton Yarn	Minimum Thickness of Code Braid, in.
0 to 0.200..	14/1 or 30/2	0.015
0.201 to 0.350..	12/1 or 26/2	0.017
0.351 to 0.800..	10/1 or 20/2	0.020
0.801 to 1.500..	12/2	0.025
1.501 to 3.000..	8/2	0.031

(c) The number of carriers and the number of ends per carrier used in applying the braid shall be optional providing all other limitations of Sec-

TABLE VIII.—MINIMUM THICKNESS OF STANDARD BRAID

Diameter Under Braid, in.	Minimum Thickness of Standard Braid, in.	Corresponding Minimum Size and Ply of Cotton Yarn
0 to 0.200.....	0.016	30/2
0.201 to 0.350.....	0.017	26/2
0.351 to 0.800.....	0.020	20/2
0.801 to 1.500.....	0.026	12/2
1.501 to 3.000.....	0.032	8/2

TABLE IX.—MINIMUM THICKNESS OF HEAVY BRAID.

Diameter Under Braid, in.	Minimum Thickness of Heavy Braid, in.	Corresponding Minimum Size and Ply of Cotton Yarn
0 to 0.200.....	0.020	20/2
0.201 to 0.300.....	0.022	16/2
0.301 to 0.600.....	0.026	12/2
0.601 to 1.000.....	0.032	8/2
1.001 to 1.500.....	0.037	6/2
1.501 to 2.000.....	0.045	4/2
2.001 to 3.000.....	0.056	4/3

tions 30 and 31 are fulfilled. The picks per inch shall be not less than the value as calculated by the following formula:

$$P = \sqrt{\left[ \frac{1}{tE + M} \right]^2 - \left[ \frac{C}{2\pi(D + T)} \right]^2}$$

where:

$P$  = the picks per inch,

$T$  = the thickness of braid, in inches,

$$T = 2t$$

$t$  = the thickness of one end of cotton, in inches =  $\frac{0.0279 K}{\sqrt{S}}$

$K$  = cabling factor for plied yarn,

values of  $K$  are:  $\begin{cases} 2 \text{ ply} = 1.60 \\ 3 \text{ ply} = 2.00 \\ 4 \text{ ply} = 2.36 \end{cases}$

$S$  = the cotton ply, size or number,  
 $E$  = the number of ends per carrier,  
 $C$  = the number of carriers,  
 $D$  = the diameter under braid, in inches,

$M$  = the space between each set of carrier threads, in inches,

$$M = X (0.011 + 7.0 t^2 E^2)$$

$X$  = the closeness constant:

Values of $X$		
Diameter Under Braid, in.	Code Braids	Standard and Heavy Braids
0 to 0.500.....	1.7	1.1
0.501 and over.....	1.5	1.0

For convenience in calculating the more usual braid constructions, the formula for picks may be reduced to:

$$P = \sqrt{Y - \frac{Z}{(D + T)^2}}$$

where:

$$Y = \frac{1}{(tE + M)^2}$$

and

$$Z = \left(\frac{C}{2\pi}\right)^2$$

Values of  $Y$  for some of the more commonly used cottons are given in Tables X and XI.

TABLE X.—VALUES OF  $Y$  FOR CODE BRAIDS.

Size and Ply of Cotton Yarn	Thickness of Braid, $T$	Values of $Y$ for Code Braids									
		For Diameters Under 0.500 in.					For Diameters 0.501 in. and Over				
		2 Ends	3 Ends	4 Ends	5 Ends		2 Ends	3 Ends	4 Ends	5 Ends	6 Ends
14/1	0.015	755	453	...	...		...	...	...	...	...
12/1	0.0161	699	406	...	...		...	...	...	...	...
10/1	0.0176	625	351	212	136		...	...	...	...	...
8/1	0.0197	541	290	170	107	603	325	191	120	...	...
30/2	0.0162	693	397	246	161	...	...	...	...	...	...
28/2	0.0168	662	377	230	149	...	...	...	...	...	...
26/2	0.0175	631	355	215	138	...	...	...	...	...	...
24/2	0.0182	601	333	199	127	...	...	...	...	...	...
22/2	0.0190	568	310	183	115	...	...	...	...	...	...
20/2	0.0200	528	284	166	103	603	318	186	116	77	...
18/2	0.0210	497	260	150	92	563	292	168	103	68	...
16/2	0.0223	452	232	133	81	516	262	149	91	59	...
14/2	0.0238	412	207	115	69	463	232	129	78	50	...
12/2	0.0257	367	176	97	57	411	199	109	65	41	...
10/2	0.0282	316	148	79	46	353	166	88	52	32	...
8/2	0.0316	262	108	60	34	292	130	68	39	24	...
16/3	0.0278	322	152	81	47	362	170	91	53	33	...
14/3	0.0298	288	132	69	40	324	148	78	45	28	...
12/3	0.0322	252	112	57	32	282	126	65	37	22	...
10/3	0.0352	212	91	46	25	238	103	52	29	17	...
8/3	0.0394	171	70	34	18	191	79	39	21	13	...

Values of  $Z$  for the standard braiders are:

Number of Carriers	Values of $Z$
12.....	3.65
16.....	6.49
20.....	10.13
24.....	14.59
36.....	32.80
48.....	58.40

(d) For braid over twin wires use 1.64 times the conductor diameter to determine the proper braid.

(e) When two braids are specified, the outer braid shall be determined by the diameter over the inner braid. When the diameter change-over limit is such that Table VIII or Table IX and this section require different sizes of yarn on the inner and outer braids, both braids may be of the size of yarn as required on the outer braid.

(f) When two braids are applied in one operation, these specifications shall apply to the inner and outer braids except that picks per inch and angle of braid for inner braid shall be governed by the construction of the machine.

(g) These specifications do not apply to braids for fixture wire or to fancy or special braids.

TABLE XI.—VALUES OF  $Y$  FOR STANDARD AND HEAVY BRAIDS.

Size and Ply of Cotton Yarn	Thickness of Braid, $T$	Values of $Y$ for Standard and Heavy Braids															
		For Diameters Under 0.500 in.								For Diameters 0.501 in. and Over							
		2 End	3 End	4 End	5 End					2 End	3 End	4 End	5 End	6 End	7 End	8 End	
30/2	0.0163	1081	592	358	233	...	...	...	...	...	...	...	...	...	...	...	...
26/2	0.0175	982	526	312	220	...	...	...	...	...	...	...	...	...	...	...	...
24/2	0.0182	930	492	290	185	...	...	...	...	...	...	...	...	...	...	...	...
20/2	0.0200	807	416	241	151	878	446	259	163	...	...	...	...	...	...	...	...
16/2	0.0223	683	339	193	118	738	365	207	127	...	...	...	...	...	...	...	...
14/2	0.0238	616	302	168	103	664	324	180	111	...	...	...	...	...	...	...	...
12/2	0.0258	538	257	142	85	582	276	152	92	59	40	28	...	...	...	...	...
10/2	0.0282	...	...	...	...	497	231	125	74	48	32	22	...	...	...	...	...
8/2	0.0316	...	...	...	...	407	183	96	56	35	23	16	...	...	...	...	...

(h) In Tables XII and XIII are tabulated the required picks per inch as calculated by the formula in Paragraph (c) and the later by corresponding braid angle for braids applied with the minimum size and ply of cotton, and a definite number of ends per carrier. However, it is not

TABLE XII.—TYPICAL CONSTRUCTIONS FOR CODE BRAID.

Nominal Diameter Over Insulation, In.	Car- riers	Picks per Inch																			
		14/1		30/2		26/2 and 12/1		24/2		20/2 and 10/1				16/2		12/2		10/2		8/2	
		2	3	2	3	2	3	2	3	2	3	4	2	3	4	2	3	4	3	4	
		Size and Ply....		Ends....																	
0.151 to 0.175.....	16.....	23.4	15.8	22.2	13.9	20.8	12.5	20.1	11.6	18.5											
	20.....	20.8	12.5	19.4	12.4	17.8		17.0		15.0											
	16.....	24.4	17.2	23.2	15.5	21.8	14.1	21.2	13.4	19.5											
	20.....	22.5	14.4	21.2	12.4	19.7		16.1		13.8											
	24.....	20.1		18.5		16.8		15.2	21.9	14.5	20.2	12.8									
0.176 to 0.200.....	16.....	22.5				21.0	15.2	20.3	12.0	18.5											
	20.....	20.0				18.9		18.1		16.1											
	16.....	24.4				23.1	16.0	22.4	15.3	20.7											
	20.....	21.8	14.2	21.2	13.4	19.4	11.5														
	24.....	20.2	11.4	19.4	10.5	17.6		19.4	12.6	17.0											
0.201 to 0.225.....	16.....	23.6	16.7	23.1	16.0	21.3	14.5			19.4											
	20.....	22.6	15.4	22.0	14.7	20.3	13.0			18.4											
	16.....	24.4	17.3	23.4	16.7	21.7	15.1			20.0											
	20.....	23.4	16.4	22.7	15.7	21.0	14.1			19.2											
	24.....	22.5	15.2	21.9	14.5	20.2	12.7			18.2											
0.226 to 0.250.....	16.....									20.3											
	20.....									18.0											
	16.....									17.7											
	20.....									20.5											
	24.....									18.2											
0.251 to 0.300.....	16.....									18.0											
	20.....									16.9											
	16.....									18.1											
	20.....									20.0											
	24.....									17.4											
0.301 to 0.360.....	16.....									19.5											
	20.....									22.6											
	16.....									22.1											
	20.....									18.3											
	24.....									19.8											
0.361 to 0.400.....	16.....									15.0											
	20.....									16.9											
	16.....									15.2											
	20.....									13.0											
	24.....									12.4											
0.401 to 0.450.....	16.....									14.7											
	20.....									17.3											
	16.....									17.0											
	20.....									12.9											
	24.....									15.3											
0.451 to 0.500.....	16.....									13.4											
	20.....									13.1											
	16.....									13.7											
	20.....									13.5											
	24.....									13.1											
0.501 to 0.600.....	16.....									12.4											
	20.....									12.4											
	16.....									13.4											
	20.....									13.1											
	24.....									12.4											
0.601 to 0.800.....	16.....									9.9											
	20.....									9.9											
	16.....									12.4											
	20.....									12.2											
	24.....									9.1											
1.001 to 1.200.....	16.....									12.5											
	20.....									12.5											
	16.....									13.1											
	20.....									12.4											
	24.....									13.8											
1.201 to 1.500.....	16.....									10.1											
	20.....									8.9											
	16.....									13.5											
	20.....									13.5											
	24.....									9.6											
1.501 to 2.000.....	16.....									10.0											
	20.....									11.6											
	16.....									11.2											
	20.....									8.0											
	24.....									7.6											
2.001 to 3.000.....	16.....									11.0											
	20.....									10.7											
	16.....									7.1											
	20.....									8.1											
	24.....									8.0											
	16.....									7.7											
	20.....									7.7											
	16.....									7.7											
	20.....									7.7											
	24.....									7.7											

the intent of these specifications to limit the braids to the scopes of Tables XII and XIII since heavier cottons or a variation in ends per carrier are permissible providing all requirements of Sections 30 and 31 are fulfilled.

(c) *Picks per Inch*, on a 4-in. sample after wiping off outside of braid with chloroform.

(d) *Angle of Braid* by formula:

$$\text{Tangent of Angle} = \frac{2\pi P(D + T)}{C}$$

TABLE XIII.—TYPICAL CONSTRUCTIONS FOR STANDARD AND HEAVY BRAID.

Diameter Under Braid, <i>D</i> , in.	Size and Ply of Cotton Yarn, min.	Number of Ends	Thick- ness of Braid, min., in.	Diamet- er Mean Avg. ( <i>D</i> + <i>T</i> ), in.	12- Carrier Braiders		16- Carrier Braiders		20- Carrier Braiders		24- Carrier Braiders		36- Carrier Braiders		48- Carrier Braiders	
					Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.	Picks per inch	Braid Angle, deg.
STANDARD BRAID																
0.091 to 0.110	30/2	2	0.0163	0.106	27.7	57.0	22.6	43.0								
0.111 to 0.125	30/2	2	0.0163	0.116	28.6	60.0	24.7	48.0								
0.126 to 0.150	30/2	2	0.0163	0.134	29.8	65.0	27.0	55.0	23.0	44.0						
0.151 to 0.175	30/2	2	0.0163	0.154	30.6	68.0	28.6	60.0	25.8	51.0						
0.176 to 0.200	30/2	2	0.0163	0.179	31.2	71.0	29.8	64.0	27.8	58.0						
0.201 to 0.225	30/2	2	0.0163	0.204	31.6	73.0	30.6	68.0	29.1	62.0						
0.226 to 0.250	26/2	2	0.0175	0.231	30.2	74.3	29.3	69.4	28.1	63.9	26.6	58.1				
0.251 to 0.300	26/2	2	0.0175	0.256	30.4	76.2	29.7	71.5	28.8	66.7	27.6	61.6				
0.301 to 0.350	26/2	2	0.0175	0.293	30.4	78.0	30.2	74.0	29.5	70.0	28.6	65.0				
0.351 to 0.400	20/2	3	0.020	0.343					30.0	73.0	29.4	69.0				
0.401 to 0.450	20/2	3	0.020	0.396					18.8	66.0	18.0	62.0				
0.451 to 0.500	20/2	3	0.020	0.445					19.1	70.0	18.5	65.0				
0.501 to 0.600	20/2	3	0.020	0.495					19.4	72.0	18.9	68.0				
0.601 to 0.800	20/2	3	0.020	0.570					20.4	75.0	20.1	72.0				
0.801 to 1.000	20/2	3	0.020	0.720					20.6	78.0	20.5	76.0				
1.001 to 1.200	12/2	4	0.0258	0.925					11.8	73.7	11.6	70.3	10.7	59.8	9.2	48.6
1.201 to 1.500	12/2	4	0.0258	1.125					12.0	76.8	11.9	74.0	11.2	65.7	10.3	56.0
1.501 to 2.000	12/2	4	0.0258	1.376							12.0	77.0	11.6	70.2	11.0	63.2
2.001 to 3.000	8/2	5	0.0316	1.782							7.2	73.3	6.8	64.6	6.1	55.0
	8/2	5	0.0316	2.532							7.3	78.2	7.1	72.2	6.9	66.2
HEAVY BRAID																
0.091 to 0.110	20/2	2	0.020	0.110	22.6	52.5	16.5	35.5								
0.111 to 0.125	20/2	2	0.020	0.120	23.6	56.0	18.9	41.7								
0.126 to 0.150	20/2	2	0.020	0.138	24.6	58.3	21.5	49.3								
0.151 to 0.175	20/2	2	0.020	0.158	25.7	64.6	23.3	55.3	20.0	44.8						
0.176 to 0.200	20/2	2	0.020	0.183	26.4	67.5	24.8	60.5	22.7	52.5	19.2	42.5				
0.201 to 0.225	20/2	2	0.020	0.208	26.9	70.4	25.5	65.8	23.7	57.0	21.6	49.5				
0.226 to 0.250	16/2	2	0.0223	0.235			23.8	56.5	22.4	58.8	20.4	51.5				
0.251 to 0.300	16/2	2	0.0223	0.260			25.8	77.8	23.0	62.0	21.6	55.6				
0.301 to 0.350	16/2	3	0.0223	0.297			16.3	62.3	15.0	54.5	13.2	49.5				
0.351 to 0.400	12/2	3	0.0258	0.351					13.2	55.4	11.8	47.3				
0.401 to 0.450	12/2	3	0.0258	0.401					13.9	60.1	12.9	53.5				
0.451 to 0.500	12/2	3	0.0258	0.451					14.4	63.9	13.6	58.1				
0.501 to 0.600	12/2	3	0.0258	0.501					14.7	66.6	14.1	62.0				
0.601 to 0.800	8/2	3	0.0258	0.576							15.2	66.4	13.3	55.7		
0.801 to 1.000	8/2	3	0.0316	0.732							12.5	67.3	11.1	57.2		
1.001 to 1.200	6/2	2	0.0316	0.932							12.9	72.3	12.0	65.0	10.8	53.0
1.201 to 1.500	6/2	2	0.037	1.137									17.0	74.9	16.4	67.7
1.501 to 2.000	4/2	3	0.037	1.387									17.2	77.7	16.8	71.8
2.001 to 3.000	4/3	3	0.0446	1.795									8.6	71.3	8.1	62.3
	4/3	3	0.0556	2.556									6.6	72.8	6.3	64.5

### Measurements

32. The various quantities shall be determined as follows:

(a) *Diameter Under Braid*, by a suitable micrometer reading.

(b) *Number of Carriers and Ends per Carrier*, by examination of braid.

(c) *Thickness of Braid*.—A sample of braid approximately 2 in. in length shall be carefully slit and removed from the completed wire and immersed in chloroform or other suitable solvent for 5 min. The solvent shall be decanted and the operation repeated until the cotton

is substantially free of saturating and finishing material. The sample shall then be dried by pressing between blotting papers. The extracted sample shall be measured by means of a dial micrometer having a presser foot 0.4 in. in diameter and a weight on the sample of 50 g. Five readings shall be made at different points on the sample and the average taken as the thickness of the braid. In the case of a double braid, each braid shall be removed and measured separately.

#### Number of Tests

33. Samples shall be taken at the option of the purchaser.

#### FINISH

##### Saturation and Finish of Braids

34. The braid or braids on wires and cables for indoor use shall be saturated and finished in accordance with the requirements specified in Sections 35 to 38, inclusive. The braid or braids on wires and cables for outside aerial use shall be saturated and finished in accordance with the requirements specified in Sections 39 to 44, inclusive.

##### *Finish for Indoor Use*

##### Saturant and Finish

35. The braid covering on single conductor 600-v. wires and cables for indoor use shall be thoroughly saturated with a flame-retardant, moisture-resistant compound. On all other wires and cables for indoor use, the braid covering shall be thoroughly saturated with a moisture-resistant compound. The surface of a finished, braid-covered wire or cable for indoor use shall be smooth and free from tackiness.

##### Horizontal Flame Test

36. The flame-retardant properties of fibrous coverings, other than tapes, on

single conductor 600-v. wires, not smaller than No. 14 A.w.g., shall be such that the flaming section of a specimen subjected to the horizontal flame test shall not extend in either direction for a distance greater than 2 in. from the center point of the application of the test flame after the flame has been applied for a period of 30 sec.

##### Moisture Test

37. The moisture-resistant properties of the braid covering on wires and cables for indoor use shall be such that the saturated and finished braid covering will not absorb more than 20 per cent of its weight of moisture after immersion in water at 21 C. for a period of 24 hr.

##### Bend Test

38. The flexibility of the finished braid covering on wires and cables for indoor use shall be such that when a sample of wire or cable is bent around a mandrel of the diameter prescribed, and in the manner specified in Section 36 (b) of Tentative Methods D 470,<sup>4</sup> no threads of the braid covering shall be broken. The finishing material shall not flake off the wire or cable without actual rubbing of the surface. Cracking of the finish shall not be considered cause for rejection.

##### *Finish for Outside Aerial Use*

##### Saturant

39. The saturant used in the braid covering on wire or cable for outside aerial use shall be an asphaltic-base material, or an asphaltic-base material blended with suitable high-melting-point waxes, but in neither case shall any volatile materials such as paraffin scale wax, wax tailings, or flux oils be added. The physical characteristics of the saturant, before being placed in the saturating tank, shall conform to the

requirements prescribed in the following table:

	Limit	A.S.T.M. Designation <sup>a</sup>
Softening point (ring-and-ball method), deg. Fahr. ....	145 min.	D 36
Penetration at 25 C. (77 F.), 100 g., 5 sec. ....	20 to 35	D 5
Loss on heating at 325 F. (163 C.), 50 g., 5 hr., per cent. ....	0.3 max.	D 6
Changes after loss on heating test (crust formed on specimen during heating test removed):		
Softening point (Note) (ring-and-ball method), max. rise. ....	10 F.	D 36
Penetration at 25 C. (77 F.), 100 g., 5 sec. ....		
Max. change. ....	10	D 5
Min. penetration. ....	15	D 5

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Softening Point of Bituminous Materials (Ring-and-Ball Method) (A.S.T.M. Designation: D 36).<sup>3</sup>

Standard Method of Test for Penetration of Bituminous Materials (A.S.T.M. Designation: D 5).<sup>3</sup> and

Tentative Method of Test for Loss on Heating of Oil and Asphaltic Compounds (A.S.T.M. Designation: D 6).<sup>3</sup>

Note.—The softening point after the loss on heating test shall be determined in water if the test before heating was made in water, or in glycerol if the test before heating was made in glycerol.

### Saturation

40. Saturation of the braid covering with the saturant specified shall be so thorough that no white or unsaturated ends of fiber will be exposed by a diagonal cut through the covering.

### Finish

41. (a) The saturated braid covering of the wire or cable for outside aerial use shall be finished with a dark colored flexible coating material (except as otherwise specified), such as stearine pitch or high-softening-point asphalt, and coated with mica flake or other suitable finely divided dry nonconducting material, to provide a smooth surface free of tackiness and conforming to the bending, melting, and dripping requirements prescribed in Sections 42 to 44, inclusive. Excess mica shall be avoided.

(b) The outer braid covering of the finished wire or cable, together with the compounds in and over this covering, shall contain not less than 5 per cent nor more than 20 per cent by weight,

of well-dispersed, nonmetallic commercial 90 per cent, 325-mesh, slate dust, or other finely divided dry mineral filler as agreed upon by the manufacturer and the purchaser.

(c) If the cable construction involves two or more over-all braid coverings, the inner covering may contain the mineral filler specified in Paragraph (b), but in no case shall the complete covering exceed a mineral filler content of 15 per cent by weight.

### Bend Test

42. (a) Each specimen of finished wire or cable for the bending test shall be wrapped around a mandrel having an over-all diameter equal to the cable diameter multiplied by the factor given in Table XIV.

TABLE XIV.—FACTORS FOR DETERMINING BENDING TEST MANDREL DIAMETER.

Cable Diameter, in.	Covered Size, A.w.g. numbers	Multiplying Factors	
		Conductor Insulation $\frac{1}{4}$ in. and under in Thickness	Conductor Insulation $\frac{1}{4}$ in. to $\frac{1}{2}$ in. Thickness
0 to 0.5.....	Any size.....	3	4
0.501 to 1.000..	No. 0000 and under	4	5
	No. 0000 and over..	6	7
1.001 to 1.500..	No. 0000 and under	6	7
	No. 0000 and over..	8	10
Over 1.500.....	Any size.....	12	12

(b) Single conductor cable 0.500 in. and under in diameter shall be wrapped three times around the mandrel, single conductor cable over 0.500 in. in diameter and any multiple conductor cable shall be wrapped once around the mandrel.

(c) The specimens shall be maintained at a temperature of 32 F. (0 C.) for approximately 1 hr. before bending, and bent at a uniform rate not greater than 12 turns per minute, immediately upon removal from the cooling chamber.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

(d) The yarn in the fibrous covering shall not break under test nor shall the finishing material flake off the wire or cable without actual rubbing of the surface. Cracking of the finish shall not be considered cause for rejection.

### Melt Test

43. A 6-in. specimen of the finished wire or cable shall be wrapped, except for 1 in. at each end, with a piece of clean white glazed paper. The wrapped specimens shall be suspended horizontally by the ends, in an electrically-heated oven, equipped with suitable automatic temperature control, which has been previously heated to 148 to 152 F. (64.4 to 66.7 C). The wire or cable shall be maintained at this temperature for 30 min., and then allowed to cool before inspection. After this test, the paper shall not adhere to the cable, nor shall the compound have become sufficiently fluid to be transferred to the paper to form a ridge perceptible to the touch.

### Drip Test

44. Specimens of finished wire or cable for the drip test shall be cut in 6-in. lengths, and the wire bared for a distance of  $\frac{3}{4}$  in. from each end. The specimens, with one end secured in a suitable clamp, shall be suspended at an angle of 45 deg. in an electrically-heated oven, equipped with suitable automatic temperature control, which has been previously heated to 178 to 182 F. (81.1 to 83.3 C.), and the wire maintained at this temperature for 1 hr. At the end of this time the compound shall not have dripped, formed globules on the lower side of the specimen, or formed bubbles upon its surface.

### Methods of Testing

45. The properties for finish enumerated in these specifications shall be de-

termined in accordance with the procedures described in Sections 33 to 39 of the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470).<sup>4</sup>

## LEAD SHEATHS

### Composition

46. (a) Lead sheaths shall consist of chemical lead, acid lead, copper lead, or common desilverized lead A as prescribed in the Standard Specifications for Pig Lead (A.S.T.M. Designation: B 29).<sup>3</sup>

(b) Lead sheaths shall be tightly formed around the core of the wire or cable.

### Thickness

47. Lead sheaths shall have an average thickness not less than that indicated in Table XV and the minimum thickness shall in no place be less than 90 per cent of the required average thickness.

TABLE XV.—THICKNESS OF LEAD SHEATHS.

Diameter of Core, in. <sup>a</sup>	Thickness of Sheath, sixty-fourths of an inch
Over 3.000.....	9
2.001 to 3.000.....	8
1.501 to 2.000.....	7
1.051 to 1.500.....	6
0.701 to 1.050.....	5
0.426 to 0.700.....	4
0.000 to 0.425.....	3

<sup>a</sup> For twin cables, use the major axis.

### Measurement

48. A short section of the lead sheath shall be removed from the wire or cable and the thickness measured with a suitable micrometer caliper at not less than ten points, of approximately equal spacing, care being taken to include the thinnest and the thickest parts.

## INSPECTION AND REJECTION

### Inspection and Test

49. (a) Inspection shall be made prior to shipment and at the place of manufacture.



(b) The manufacturer shall notify the purchaser sufficiently in advance of the completion of the wire or cable to permit of arrangements for inspection.

(c) The inspector representing the purchaser shall have free entry at all times, while work on the contract of the purchaser is being performed, to all parts of the manufacturer's works which concern the manufacture of the wire ordered, except compounding room. The manufacturer shall afford the inspector, without charge, all reasonable facilities to satisfy him that the wire is being furnished in accordance with these specifications.

(d) The purchaser at his option may make the various tests on samples in his own laboratory or elsewhere, but such

tests shall be made at the expense of the purchaser.

#### Rejection

50. Each coil, reel, or length which fails to conform to the physical and electrical requirements of these specifications shall be rejected.

#### Rehearing

51. Samples of rejected material shall be preserved for two weeks from the date of the test report. In case of dissatisfaction with the results of the test, the manufacturer may make claim for a rehearing within that time. Sealed duplicate samples may be retained by the manufacturer at his option.

(See Appendices, p. 1119)

## APPENDIX I

ANNEALED COPPER STANDARD<sup>a</sup>

The following shall be taken as normal values for standard annealed copper:

(a) *Resistance*.—At a temperature of 20 C. the resistance of a wire of standard annealed copper 1 m. in length and of a uniform section of 1 sq. mm. is  $\frac{1}{8}$  ohm = 0.017241 ohm.

(b) *Density*.—At a temperature of 20 C. the density of standard annealed copper is 8.89 g. per cu. cm.

(c) *Temperature Coefficient of Resistance*.—At a temperature of 20 C. the "constant mass" temperature coefficient of resistance of standard annealed copper, measured between two poten-

tial points rigidly fixed to the wire, is 0.00393 = 1/254.45 per deg. Cent.

(d) Other values of resistivity of standard annealed copper at 20 C. deduced from Paragraphs (a) and (b) are as follows:

Resistance of a wire of uniform section, 1 meter long and weighing 1 g. = 0.15328 ohm.

Resistance of a wire with a uniform section of 1 cir. mil and 1 ft. long = 10.371 ohm.

Resistance between opposite faces of 1 cu. cm. = 1.7241 microhm.

Resistance between opposite faces of 1 cu. in. = 0.67879 microhm.

## APPENDIX II

## INTERPRETATION OF INSULATION THICKNESSES IN TABLE II

For series-street-lighting cable, use the following procedure:

A1. (a) If the transformer is not equipped with protectors, use the open circuit secondary voltage of the transformer as rated voltage of the cable and select the proper wall thickness from Table II.

(b) If the transformer is equipped with protectors, use the closed or loaded secondary voltage of the transformer as the rated circuit voltage of the cable and select the proper wall thickness from Table II.

NOTE.—It is preferable, both in Paragraphs (a) and (b) above, to select a cable with a higher voltage rating than obtained using the above procedure, both as a measure of safety and to allow the substitution of a larger transformer without necessitating the replacement of the cable.

The thickness of insulation for the various systems shall be determined as follows:

A2. (a) For three-phase systems with grounded or ungrounded neutral, use thickness values in accordance with Table II.

(b) For single or two-phase systems up to and including 5000 v., use thickness values in accordance with Table II, as specified for grounded

neutral. Where it is not definitely specified that a line operates as an isolated single or two-phase system it shall be considered as a branch of a grounded three-phase circuit and the thickness of insulation shall be that required by the phase-to-phase voltage of this grounded, three-phase circuit as given in Table II.

(c) For single or two-phase systems operating at over 5000 v. with one side grounded, multiply the circuit voltage (phase-to-phase) by 1.73 and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table II.

(d) For single or two-phase systems operating at over 5000 v. with the center grounded, multiply the circuit voltage (phase-to-phase) by 0.866 ( $\frac{1}{2}\sqrt{3}$ ) and use the resulting voltage value to select the corresponding insulation thickness for grounded neutral as specified in Table II.

(e) For single or two-phase ungrounded systems operating at over 5000 v., multiply the circuit voltage (phase-to-phase) by 0.866 ( $\frac{1}{2}\sqrt{3}$ ) and use the resulting voltage value to select the corresponding insulation thickness for ungrounded neutral as specified in Table II.

(f) For direct-current systems, consider the same as single-phase alternating-current systems in accordance with Paragraph A2 (b).

<sup>a</sup> Extracts from American Institute of Electrical Engineers' Standard No. 30 (1932).

# *Tentative Specifications for*

## INSULATED WIRE AND CABLE: HEAT-RESISTING SYNTHETIC RUBBER COMPOUND<sup>1</sup>



A.S.T.M. Designation: D 754-46a T

(Revision of Specifications D 754-46 T)

ISSUED, 1943; REVISED, JANUARY 1946, AUGUST 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These specifications cover wire and cable insulated with a vulcanized compound, the rubber hydrocarbon content of which shall consist substantially of synthetic rubber. They are intended to permit the use of the compound known as "Government Rubber," type GR-S, but are not restricted to this type. The suitability of the compound as insulation shall be determined solely by physical and electrical tests, and compounds of any vulcanized, general purpose synthetic which conforms to the test requirements shall be considered acceptable. This compound is a heat-resisting type and is ordinarily considered suitable for use at conductor temperatures not in excess of 167 F. (75 C.). This compound is not recom-

mended for installation or use in severe flexing service at a temperature lower than -35 C.

(b) Except for the insulating compound, wire and cable supplied under these specifications shall conform to the construction requirements of the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (A.S.T.M. Designation: D 27).<sup>3</sup>

### Physical Properties

2. The insulation shall consist of a properly vulcanized synthetic rubber compound which shall conform to the requirements prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min., psi.....	700
Elongation at rupture, min., per cent.....	300
Tensile strength and elongation after 20 hr. air pressure heat test, min., per cent of original value .....	50
Tensile strength and elongation after 168 hr. at 80 C. in oxygen pressure test, min., per cent of original value.....	50

NOTE 1.—The above values are applicable only to conductors having nominal wall thicknesses of insulation  $\frac{3}{32}$  in. and greater.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revisions accepted by the Administrative Committee on Standards, January 10, 1946, and September 9, 1946.

Prior to their publication as tentative, these specifications were issued as Emergency Specifications ES-33 from August 13, 1943, to December 27, 1943.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# SPECIFICATIONS FOR SYNTHETIC RUBBER INSULATED WIRE (D 754 -46a T) 1121

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For nonleaded submarine cables,  $\frac{1}{2}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits except where one side of a delta system is permanently grounded, in which case, multiply the circuit voltage (phase to phase) by 1.73 and use the resulting voltage to select the corresponding insulation thickness.

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thick- ness on Each Conductor, sixty- fourths of an inch	Test Voltage
0 to 300.....	No. 18 to No. 16.....	2	1 000
0 to 600.....	No. 14 <sup>a</sup> to No. 12 <sup>a</sup> .....	2	1 500
	No. 16 to No. 9 <sup>b</sup> .....	3	3 000
	No. 8 to No. 2.....	4	3 500
	No. 1 to No. 0000.....	5	4 000
	225 000 to 500 000 cir. mils.....	6	5 000
601 to 1000.....	525 000 to 1 000 000 cir. mils.....	7	6 000
	Over 1 000 000 cir. mils.....	8	7 000
	No. 14 to No. 8.....	4	5 000
	No. 7 to No. 2.....	5	6 000
	No. 1 to No. 0000.....	6	7 500
1001 to 2000.....	225 000 to 500 000 cir. mils.....	7	9 000
	525 000 to 1 000 000 cir. mils.....	8	10 000
	Over 1 000 000 cir. mils.....	9	11 000
	No. 14 to No. 8.....	5	6 000
	No. 7 to No. 2.....	6	7 500
2001 to 3000.....	No. 1 to No. 0000.....	7	9 000
	225 000 to 500 000 cir. mils.....	8	10 000
	525 000 to 1 000 000 cir. mils.....	9	11 000
	Over 1 000 000 cir. mils.....	10	12 500
	No. 10 to No. 8.....	7	9 000
3001 to 4000.....	No. 7 to No. 2.....	8	10 000
	No. 1 to No. 0000.....	9	11 000
	225 000 to 500 000 cir. mils.....	10	12 500
	525 000 to 1 000 000 cir. mils.....	11	15 000
	Over 1 000 000 cir. mils.....	12	17 500
4001 to 5000.....	No. 8.....	10	12 500
	No. 7 to No. 2.....	10	12 500
	No. 1 to No. 0000.....	10	12 500
	225 000 to 500 000 cir. mils.....	11	13 500
	525 000 to 1 000 000 cir. mils.....	11	13 500
5001 to 6000 (grounded).....	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	10	12 500
	225 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	12	15 000
5001 to 6000 (ungrounded).....	225 000 to 1 000 000 cir. mils.....	12	15 000
	Over 1 000 000 cir. mils.....	13	16 500
	No. 8 to No. 0000.....	11	13 500
	225 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
6001 to 7000 (grounded).....	No. 8 to No. 0000.....	14	17 500
	225 000 to 1 000 000 cir. mils.....	14	17 500
	Over 1 000 000 cir. mils.....	15	19 000
	No. 8 to No. 0000.....	12	15 000
	225 000 to 1 000 000 cir. mils.....	12	15 000
6001 to 7000 (ungrounded).....	Over 1 000 000 cir. mils.....	13	16 500
	No. 8 to No. 0000.....	16	20 000
	225 000 to 1 000 000 cir. mils.....	16	20 000
	Over 1 000 000 cir. mils.....	17	21 000
	No. 8 to No. 0000.....	16	20 000
7001 to 8000 (grounded).....	225 000 to 1 000 000 cir. mils.....	16	20 000
	Over 1 000 000 cir. mils.....	17	21 000
	No. 8 to No. 0000.....	16	20 000
	225 000 to 1 000 000 cir. mils.....	16	20 000
	Over 1 000 000 cir. mils.....	17	21 000

<sup>a</sup> Building wire with  $\frac{3}{4}$ -in. wall is permitted in sizes 14 and 12 by the National Electrical Code and will be furnished unless otherwise specified.

<sup>b</sup> Sizes Nos. 16 and 18 are not recognized by the National Electrical Code for 600-v. service.

NOTE 2.—When the insulation has been subjected to a second vulcanization, such as occurs when used as the insulation of single conductors under vulcanized jackets, the physical properties shall be not less than 75 per cent of the above requirements.

### High Voltage Test

3. Each coil, reel, or length of wire or cable, after vulcanization of the insulation shall successfully withstand, for a period of 5 min., the application of an alternating-current voltage of not less than that prescribed in Table II. Insulation less than  $\frac{1}{32}$  in. in thickness shall not be tested for electrical properties in water. At the option of the purchaser, a test of 500 v. may, however, be applied between the conductors and shield, or both, in the completed cable.

### Insulation Resistance

4. (a) Each coil, reel, or length of wire or cable after being subjected to the high-voltage test specified in Section 3, shall have an insulation resistance in megohms per 1000 ft. at a temperature of 60 F. (15.6 C.) of not less than the value of  $R$  as calculated from the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where:

$R$  = insulation resistance in megohms per 1000 ft.,

$K$  = constant of 2000,

$D$  = diameter over insulation, and

$d$  = diameter under insulation.

(b) When the temperature at which the insulation resistance is measured differs from 60 F. (15.6 C.) the resistance shall be converted to that at 60 F. (15.6 C.) by multiplying the measured value by a coefficient to be furnished by the manufacturer, corresponding to the temperature at which the measurement is made.

### Thickness

5. (a) The average thickness of the insulation shall be not less than that prescribed in Table II. The minimum thickness shall be not less than 95 per cent of the thickness required by Table II for insulations less than  $\frac{1}{8}$  in. in thickness and not less than 90 per cent of the specified thickness for insulations of  $\frac{1}{8}$  in. and greater.

(b) The thicknesses prescribed in Table II apply to single-conductor cable and the individual conductors of multiple-conductor cables, leaded or braided, except special applications such as aerial, nonleaded submarine, and vertical riser cable.

NOTE.—In Appendix II of A.S.T.M. Specifications D 27<sup>8</sup> will be found an interpretation of the insulation thicknesses given in Table II.

(c) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

(d) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement

made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement shall be made after slicing off the heavier side of the insulation.) In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

### Workmanship

6. (a) The insulation shall be homogeneous in character, tough, elastic, and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(b) *Shielding*.—Wire and cables not supported on insulators shall be shielded or suitably protected against corona in accordance with the detailed recommendations contained in Sections 20 to 24, inclusive, of A.S.T.M. Specifications D 27.<sup>3</sup>

(c) *Covering*.—The insulation of braided wires up to No. 7 A.w.g., inclusive, shall be covered with a single cotton braid. On larger sizes the fibrous covering shall consist of a cable tape and cotton braid or two cotton braids. The cable tape may be applied before vulcanization. In multiple-conductor

cables the individual conductors shall not be braid covered unless specified.

(d) *Repairs or Joints*.—When repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the

limitations on the thickness specified in Section 5.

### Rejection

7. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

(b) When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

(c) Each coil, reel, or length which fails to conform to the electrical requirements prescribed in these specifications shall be rejected.

### Methods of Testing

8. All measurements and tests necessary for determining the conformity of the insulated wire and cable with these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470),<sup>3</sup> except as otherwise prescribed in these specifications.

*Tentative Specifications for*  
**INSULATED WIRE AND CABLE: OZONE-RESISTANT  
TYPE INSULATION<sup>1</sup>**



**A.S.T.M. Designation: D 574 - 46 T**

ISSUED, 1940; REVISED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Scope**

1. (a) These specifications cover wire and cable insulated with a vulcanized rubber or rubber-like compound known commercially as ozone-resistant type insulation. This type of insulation is suitable for use for all sizes and classes of single and multiple conductor cables which are to be used for the transmission and distribution of electrical energy, either underground, submarine, aerial, or indoor power cable, under normal conditions of installation, and operating at not over 17,000 v. between phases on an ungrounded neutral system or 28,000 v. between phases on a grounded neutral system.

(b) Except for the insulation, cable supplied under these specifications, unless otherwise specified by the purchaser, shall conform to the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea

Rubber Compound (A.S.T.M. Designation: D 27).<sup>3</sup>

**Information to be Supplied by Purchaser**

2. When requesting proposals from cable manufacturers, the prospective purchaser shall furnish certain information by filling out a form similar to the following:

A. Characteristics of system on which cable is to be used:

(1) Kind of current, alternating (a. c.), or direct (d. c.).

(2) Frequency, cycles.

(3) Normal operating voltage between phases.

(4) Number of phases and conductors. If series lighting, give open circuit voltage and if system is operated with or without protectors.

(5) Whether operated with neutral solidly grounded, grounded through resistance or reactance, or ungrounded. If grounded through resistance or reactance, state the amount of same.

(6) Minimum temperature at which cable will be installed.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Administrative Committee on Standards, September 9, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



(7) Description and conditions of installations:

- (a) Ambient temperature,
- (b) Number of loaded cables in duct bank,
- (c) Load factor, and
- (d) Method of bonding and grounding of lead sheath.

B. Description and quantities of cable upon which quotations are desired:

- (1) Number of feet.
- (2) Type of cable:
  - (a) Description, such as single conductor, two-conductor flat, two-conductor round, etc.
  - (b) Rated circuit voltage, phase-to-phase,
  - (c) Size of conductors, A.w.g. number or circular mils. If conditions require stranding other than standard, complete description should be given.
  - (d) Thickness of insulation in sixty-fourths of an inch,
  - (e) Type of outer coverings, and
  - (f) Maximum allowable over-all diameter in inches. When duct space is not limited, it is desirable not to restrict the over-all diameter.

### Conductors

3. Conductors shall conform to requirements prescribed in the Tentative Specifications D 27.<sup>3</sup> For stranded conductors insulated for over 1000 v., class C stranding as prescribed in Table II of the Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (A.S.T.M. Designation: B 8) of the American Society for Testing Materials<sup>4</sup> is recommended.

### Methods of Testing

4. All measurements and tests necessary for determining the conformity of the insulated wire and cable with these specifications shall be made in accord-

ance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470)<sup>3</sup> except as otherwise prescribed in these specifications.

### Physical Properties

5. The insulation shall consist of a properly vulcanized rubber or rubber-like compound which shall conform to the requirements as to physical properties prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min. psi.....	450
Elongation at rupture, min., per cent.....	250
Set in 2-in. gage length, max., in.....	$\frac{1}{2}$
Tensile strength after 168 hr. air oven test at 157 to 159 F. (69.4 to 70.6 C.), min., psi.....	400
Elongation at rupture after 168 hr. air oven test at 157 to 159 F. (69.4 to 70.6 C.), min., per cent.....	200
Tensile strength after 48 hr. in oxygen pressure test at 157 to 159 F. (69.4 to 70.6 C.), min., psi.....	400
Elongation at rupture after 48 hr. in oxygen pressure test at 157 to 159 F. (69.4 to 70.6 C.), min., per cent.....	200

### High Voltage Test on Entire Lengths of Cable

6. (a) *Alternating-Current Voltage.*—Each coil, reel, or length of insulated conductor after vulcanization and before the application of outer coverings other than a tape used for vulcanization, shall successfully withstand the application of an alternating-current voltage of not less than that prescribed in Table II or III for a period of 5 min.

(b) *Direct-Current Voltage.*—Each coil, reel, or length of insulated conductor for service of 5001 v. and over, immediately after the insulation-resistance test (see Section 7) and while still immersed in water, shall successfully withstand the application of direct-current voltage of three times the alternating-current voltage for a period of 15 min.

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part I-B.

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that of the next larger size or higher rated circuit voltage.

NOTE 3.—For nonleaded submarine cables,  $\frac{1}{16}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—The conductor size given first for each circuit voltage class is the minimum size recommended for that voltage class.

Rated Circuit Voltage	Size of Conductor, A.w.g. numbers or circular mils	Insulation Thick- ness on Each Conductor, sixty- fourths of an inch		Alternating-Current Test Voltages	
		Neutral Grounded	Neutral Un- grounded	Neutral Grounded	Neutral Un- grounded
0 to 600.....	No. 14 to No. 9.....	3	3	4 500	4 500
	No. 8 to No. 2.....	4	4	6 000	6 000
	No. 1 to No. 0000.....	5	5	7 500	7 500
	225 000 to 500 000 cir. mils.....	6	6	8 500	8 500
	525 000 to 1 000 000 cir. mils.....	7	7	10 000	10 000
	Over 1 000 000 cir. mils.....	8	8	11 500	11 500
601 to 1000.....	No. 14 to No. 8.....	4	4	6 000	6 000
	No. 7 to No. 2.....	5	5	7 500	7 500
	No. 1 to No. 0000.....	6	6	8 500	8 500
	225 000 to 500 000 cir. mils.....	7	7	10 000	10 000
	525 000 to 1 000 000 cir. mils.....	8	8	11 500	11 500
	Over 1 000 000 cir. mils.....	9	9	13 000	13 000
1001 to 2000.....	No. 14 to No. 8.....	5	5	7 500	7 500
	No. 7 to No. 2.....	6	6	8 500	8 500
	No. 1 to No. 0000.....	7	7	10 000	10 000
	225 000 to 500 000 cir. mils.....	8	8	11 500	11 500
	Over 525 000 cir. mils.....	9	9	13 000	13 000
2001 to 3000.....	No. 10 to No. 8.....	7	7	10 000	10 000
	No. 7 to No. 0000.....	8	8	11 500	11 500
	225 000 to 1 000 000 cir. mils.....	9	9	13 000	13 000
	Over 1 000 000 cir. mils.....	10	10	14 000	14 000
3001 to 4000.....	No. 10 to No. 0000.....	9	9	13 000	13 000
	225 000 to 1 000 000 cir. mils.....	10	10	14 000	14 000
	Over 1 000 000 cir. mils.....	11	11	15 500	15 500
4001 to 5000.....	No. 8 to No. 0000.....	10	10	14 000	14 000
	225 000 to 1 000 000 cir. mils.....	11	11	15 500	15 500
	Over 1 000 000 cir. mils.....	12	12	17 000	17 000
5001 to 6000.....	No. 8 to No. 0000.....	10	12	14 000	17 000
	225 000 to 1 000 000 cir. mils.....	11	12	15 500	17 000
	Over 1 000 000 cir. mils.....	12	13	17 000	18 500
6001 to 7000.....	No. 8 to 1 000 000 cir. mils.....	11	14	15 500	20 000
	Over 1 000 000 cir. mils.....	12	15	17 000	21 000
7001 to 8000.....	No. 8 to 1 000 000 cir. mils.....	12	16	17 000	22 500
	Over 1 000 000 cir. mils.....	13	17	18 500	24 000
8001 to 9000.....	No. 6 to 1 000 000 cir. mils.....	13	17	18 500	24 000
	Over 1 000 000 cir. mils.....	14	18	20 000	25 000
9001 to 10 000.....	No. 6 to 1 000 000 cir. mils.....	14	18	20 000	25 000
	Over 1 000 000 cir. mils.....	15	19	21 000	26 500
10 001 to 11 000.....	No. 6 to 1 000 000 cir. mils.....	15	20	21 000	28 000
	Over 1 000 000 cir. mils.....	16	21	22 500	29 500
11 001 to 12 000.....	No. 6 to 1 000 000 cir. mils.....	16	22	22 500	31 000
	Over 1 000 000 cir. mils.....	17	23	24 000	32 000
12 001 to 13 000.....	No. 6 to 1 000 000 cir. mils.....	17	23	24 000	32 000
	Over 1 000 000 cir. mils.....	18	24	25 000	33 500
13 001 to 14 000.....	No. 6 to 1 000 000 cir. mils.....	18	25	25 000	35 000
	Over 1 000 000 cir. mils.....	19	26	26 500	36 000
14 001 to 15 000.....	No. 6 to 1 000 000 cir. mils.....	19	27	26 500	37 500
	Over 1 000 000 cir. mils.....	20	28	28 000	39 000
15 001 to 16 000.....	No. 4 to 1 000 000 cir. mils.....	20	28	28 000	39 000
	Over 1 000 000 cir. mils.....	21	29	29 500	40 500

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE—*Concluded.*

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.  
 NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that of the next larger size or higher rated circuit voltage.  
 NOTE 3.—For nonleaded submarine cables,  $\frac{1}{16}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.  
 NOTE 4.—The conductor size given first for each circuit voltage class is the minimum size recommended for that voltage class.

Rated Circuit Voltage	Size of Conductor, A.w.g. numbers or circular mils	Insulation Thickness on Each Conductor, sixty-fourths of an inch		Alternating-Current Test Voltages	
		Neutral Grounded	Neutral Un-grounded	Neutral Grounded	Neutral Un-grounded
16 001 to 17 000.....	No. 4 to 1 000 000 cir. mils.....	21	30	29 500	41 500
	Over 1 000 000 cir. mils.....	22	31	31 000	43 000
17 001 to 18 000.....	No. 4 to 1 000 000 cir. mils.....	22	..	31 000	.....
	Over 1 000 000 cir. mils.....	23	..	32 000	.....
18 001 to 19 000.....	No. 4 to 1 000 000 cir. mils.....	23	..	32 000	.....
	Over 1 000 000 cir. mils.....	24	..	33 500	.....
19 001 to 20 000.....	No. 2 to 1 000 000 cir. mils.....	24	..	33 500	.....
	Over 1 000 000 cir. mils.....	25	..	35 000	.....
20 001 to 21 000.....	No. 2 to 1 000 000 cir. mils.....	25	..	35 000	.....
	Over 1 000 000 cir. mils.....	26	..	36 000	.....
21 001 to 22 000.....	No. 2 to 1 000 000 cir. mils.....	26	..	36 000	.....
	Over 1 000 000 cir. mils.....	27	..	37 500	.....
22 001 to 23 000.....	No. 2 to 1 000 000 cir. mils.....	27	..	37 500	.....
	Over 1 000 000 cir. mils.....	28	..	39 000	.....
23 001 to 24 000.....	No. 2 to 1 000 000 cir. mils.....	28	..	39 000	.....
	Over 1 000 000 cir. mils.....	29	..	40 500	.....
24 001 to 25 000.....	No. 2 to 1 000 000 cir. mils.....	29	..	40 500	.....
	Over 1 000 000 cir. mils.....	30	..	41 500	.....
25 001 to 26 000.....	No. 2 to 1 000 000 cir. mils.....	30	..	41 500	.....
	Over 1 000 000 cir. mils.....	31	..	43 000	.....
26 001 to 27 000.....	No. 1 to 1 000 000 cir. mils.....	31	..	43 000	.....
	Over 1 000 000 cir. mils.....	32	..	44 500	.....
27 001 to 28 000.....	No. 1 to 1 000 000 cir. mils.....	32	..	44 500	.....
	Over 1 000 000 cir. mils.....	33	..	46 000	.....

### Insulation Resistance

7. (a) Each coil, reel, or length of insulated conductor after being subjected to the high alternating-current voltage test specified in Section 6 (a) shall have an insulation resistance in megohms per 1000 ft. at a temperature of 60 F. of not less than the value for  $R$  as calculated from the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where:

$R$  = insulation resistance in megohms per 1000 ft.,

$K$  = constant of 2000,

$D$  = diameter over insulation, on each conductor, and

$d$  = diameter over conductor.

(b) When the temperature at which the insulation resistance is measured differs from 60 F., the resistance shall be reduced to that at 60 F. by multiplying the measured value by a coefficient to be furnished by the manufacturer corresponding to the temperature at which the measurement is made.

### Double-Voltage Test on Short Samples

8. (a) Samples of cables rated at 5001 v. or more shall, when required,

be subjected to and conform to the requirements of this test.

(b) If all of the samples conform to the requirements of this test, the lot of cable to which they apply shall be considered acceptable so far as these particular requirements are concerned.

(c) If any sample fails to conform to the requirements of this test, that length of cable from which the sample was taken shall be rejected and another sample shall then be taken from each

### Cold-Bend, Long-Time Voltage Test on Short Samples

9. (a) Samples of cables rated at 5001 v. or more shall, when required, be subjected to and conform to the requirements of this test.

(b) If all of the samples conform to the requirements for this test, the lot of cable to which they apply shall be considered acceptable so far as these particular requirements are concerned.

TABLE III.—RUBBER INSULATION THICKNESSES FOR SERIES LIGHTING CIRCUITS AND ALTERNATING TEST VOLTAGES.

Open Circuit Voltage	Size of Conductor, A.w.g. numbers	Insulation Thickness on Each Conductor, sixty-fourths of an inch		Alternating-Current Test Voltages	
		Without Protectors	With Protectors	Without Protectors	With Protectors
0 to 600.....	No. 10.....	3	3	4 500	4 500
	No. 8 to No. 4.....	4	4	6 000	6 000
601 to 1000.....	No. 10 to No. 8.....		4	6 000	6 000
	No. 7 to No. 4.....		4	7 500	6 000
1001 to 2000.....	No. 10 to No. 8.....	5	4	7 500	6 000
	No. 7 to No. 4.....		5	8 500	7 500
2001 to 3000.....	No. 10 to No. 8.....	7	5	10 000	7 500
	No. 7 to No. 4.....	8	6	11 500	8 500
3001 to 4000.....	No. 10 to No. 8.....	9	7	13 000	10 000
	No. 7 to No. 4.....	9	8	13 000	11 500
4001 to 5000.....	No. 10 to No. 4.....	10	9	14 000	13 000
5001 to 6000.....	No. 8 to No. 4.....	10	9	14 000	13 000
6001 to 7000.....	No. 8 to No. 4.....	11	10	15 500	14 000
7001 to 8000.....	No. 8 to No. 4.....	12	11	17 000	15 500
8001 to 9000.....	No. 8 to No. 4.....	13	11	18 500	15 500
9001 to 10 000.....	No. 8 to No. 4.....	14	12	20 000	17 000
10 001 to 11 000.....	No. 8 to No. 4.....	15	12	21 000	17 000
11 001 to 12 000.....	No. 8 to No. 4.....	16	14	22 500	20 000
12 001 to 13 000.....	No. 8 to No. 4.....	17	14	24 000	20 000
13 001 to 14 000.....	No. 8 to No. 4.....	18	15	25 000	21 000
14 001 to 15 000.....	No. 8 to No. 4.....	19	15	26 500	21 000

of two other lengths of cable in the lot. If either such sample fails to pass this test, the lot shall be rejected. If both such second samples pass this test the lot of cable (except the length which was rejected because of failure of the first sample) shall be accepted.

(d) Failure of any cable length shall not prohibit the manufacturer from resubmitting the same length of cable for inspection, providing that it be so designated.

(c) If any sample fails to conform to the requirements for this test, that length of cable from which the sample was taken shall be rejected and another sample shall then be taken from each of two other lengths of cable in the lot of cable under test. If either such second sample fails to pass this test, the lot shall be rejected. If both such second samples pass this test, the lot of cable (except the length which was rejected

because of failure of the first sample) shall be accepted.

(d) Failure of any cable length shall not prohibit the manufacturer from re-submitting the same length of cable for inspection, providing that it be so designated.

#### Capacity and Power Factor Tests

10. After 24-hr. submersion in water at room temperature a sample of the insulated conductor shall be tested for capacity and power factor with suitable 60-cycle equipment at working voltage, and shall show a specific inductive capacity not exceeding 5.0 and a power factor not exceeding 5 per cent.

#### Ozone Resistance Test

11. (a) The samples shall show no injury to the insulation after exposure for 3 hr. to the ozone resistance test.

(b) The results of this test shall be taken as representative of the quality of each lot of cable (approximately 10,000 ft.) from which the samples were removed.

(c) If all of the samples conform to the requirements for this test, the lot<sup>5</sup> of cable to which they apply (that is, all cable submitted up to the group<sup>5</sup> containing the next set of samples, presumably 10,000 ft.) shall be considered acceptable so far as these particular requirements are concerned, unless the purchaser requests additional confirmatory tests. In that case additional samples (second samples) shall be taken from lengths designated by the purchaser, and if they pass the prescribed test, the lot of cable shall be accepted. If any second sample fails to pass this

test, the length from which it was removed shall be rejected and a third sample (or samples) shall be selected from a reel length designated by the purchaser in the same group. If all such third samples pass the prescribed test, the lot shall be accepted so far as these particular requirements are concerned but if any sample fails, the group involved shall be rejected and no more cable on that order shall be accepted unless this test has been successfully passed by samples selected from the next group or groups.

(d) If any sample of those originally selected fails to pass the prescribed test, the length from which the sample was removed shall be rejected and another sample (second sample) shall be taken from another length of cable to be designated by the purchaser and tested. If any such second sample fails to pass the prescribed test, the group involved shall be rejected. If all of such second samples pass the test, then another sample (third samples) shall be taken from a length designated by the purchaser. If all such third samples pass the prescribed test, the lot of cable which the sample represents shall be accepted (except the section which was rejected because of failure of the first sample). If any one sample fails, the group from which the samples were taken shall be rejected and no more cable on that order shall be accepted until this test has been successfully passed by samples selected from the next group or groups.

(e) Rejection of cable under this section shall not prohibit the manufacturer from resubmitting for inspection all or any part of a rejected lot of cable (except lengths from which samples were taken and which failed). However, when such cable is resubmitted, the purchaser or his representative shall be informed as to its identity.

<sup>5</sup> For the purpose of these specifications, a "group" shall consist of all cable designated as such on a list to be submitted by the manufacturer prior to the material being offered for inspection (not less than ten reel lengths in three successive working days), and a "lot" shall consist of all cable represented by the samples taken for the test prescribed (approximately 10,000 ft.).

### Thickness

12. (a) The average thickness of the insulation shall be not less than that prescribed in Table II. The minimum thickness shall be not less than 95 per cent of the thickness prescribed in Table II for insulation less than  $\frac{1}{4}$  in. in thickness and not less than 90 per cent of the specified thickness for insulation  $\frac{1}{4}$  in. and over in thickness.

(b) The thicknesses prescribed in Table II apply to single-conductor cable and the individual conductors of multiple-conductor cables, leaded or braided, except special applications such as aerial, nonleaded submarine, and vertical riser cable.

(c) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

(d) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character. The average thickness of the insulation shall be taken as one half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first

measurement is made by "slicing off" the heavier side of the insulation.) In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

### Workmanship

13. (a) The insulation shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. When the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(b) *Shielding.*—Wires and cables not supported on insulators shall be shielded or suitably protected against corona in accordance with the detailed recommendations contained in Sections 24 to 28, inclusive, of Standard Specifications D 27.<sup>3</sup>

(c) *Repairs and Joints.*—When repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 12.

### Rejection

14. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

(b) When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

Where the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested

and shall be accepted or rejected upon the results of such individual tests.

(c) Each coil, reel, or length which fails to conform to the electrical requirements of these specifications shall be rejected.



# Tentative Specifications for INSULATED WIRE AND CABLE: PERFORMANCE SYNTHETIC RUBBER COMPOUND<sup>1</sup>



**A.S.T.M. Designation: D 755 - 46a T**  
(Revision of Specifications D 755 - 46 T)

ISSUED, 1943; REVISED, 1944, JANUARY 1946, AUGUST 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. (a) These specifications cover wire and cable insulated with a vulcanized compound, the rubber hydrocarbon content of which shall consist substantially of synthetic rubber. They are intended to permit the use of the compound known as "Government Rubber," type GR-S, but are not restricted to this type. The suitability of the compound as insulation shall be determined solely by physical and electrical tests, and compounds of any vulcanized, general purpose synthetic which conforms to the test requirements shall be considered acceptable. This compound is not recommended for installation or use in severe flexing service at a temperature lower than -35 C.

(b) Except for the insulating compound, wire and cable supplied under

these specifications shall conform to the construction requirements of the Tentative Specifications for Insulated Wire and Cable: Class AO, 30 per cent Hevea Rubber Compound (A.S.T.M. Designation: D 27).<sup>3</sup>

## Physical Properties

2. The insulation shall consist of a properly vulcanized synthetic rubber compound which shall conform to the requirements prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min., psi.....	700
Elongation at rupture, min., per cent.....	300
Tensile strength after 96 hr. in oxygen pressure test, min., per cent of original value .....	75
Elongation at rupture after 96 hr. in oxygen pressure test, min., per cent of original value .....	65

NOTE 1.—The above values are applicable only to conductors having nominal wall thicknesses of insulation  $\frac{3}{32}$  in. and greater.

NOTE 2.—When the insulation has been subjected to a second vulcanization, such as occurs when it is used as the insulation of single conductors under vulcanized jackets, the physical properties shall be not less than the following:

Initial tensile strength, min., psi.....	600
Elongation at rupture, min., per cent.....	250
Tensile strength after 48 hr. in oxygen pressure test, min., psi.....	500

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revisions accepted by the Administrative Committee on Standards, January 10, 1946, and September 9, 1946.

Prior to their publication as tentative, these specifications were issued as Emergency Specifications ES-34 from August 13, 1943, to December 27, 1943.

# SPECIFICATIONS FOR SYNTHETIC RUBBER INSULATED WIRE (D 755 - 46a T) 1133

TABLE II.—THICKNESS OF INSULATION AND ALTERNATING TEST VOLTAGE.

NOTE 1.—Rated circuit voltage of alternating-current circuits is the phase-to-phase voltage.

NOTE 2.—For an intermediate size of wire or an intermediate rated circuit voltage, the thickness and the test voltage shall be that for the next larger size or higher rated circuit voltage.

NOTE 3.—For nonleaded submarine cables,  $\frac{1}{4}$  in. should be added to the specified respective walls of insulation in this table for all sizes and voltages.

NOTE 4.—Rated circuit voltages from 0 to 5000 v. in this table apply to wire for use on grounded and ungrounded circuits except where one side of a delta system is permanently grounded, in which case, multiply the circuit voltage (phase to phase) by 1.73 and use the resulting voltage to select the corresponding insulation thickness.

Rated Circuit Voltage	Size of Conductor, A. w. g. Numbers or Circular Mils	Insulation Thick- ness on Each Conductor, sixty- fourths of an inch	Test Voltage
0 to 300.....	No. 18 to No. 16.....	2	1 000
0 to 600.....	No. 14 <sup>a</sup> to No. 12 <sup>a</sup> .....	2	1 500
	No. 16 to No. 9 <sup>b</sup> .....	3	3 000
	No. 8 to No. 2.....	4	3 500
	No. 1 to No. 0000.....	5	4 000
	225 000 to 500 000 cir. mils.....	6	5 000
601 to 1000.....	525 000 to 1 000 000 cir. mils.....	7	6 000
	Over 1 000 000 cir. mils.....	8	7 000
	No. 14 to No. 8.....	4	5 000
	No. 7 to No. 2.....	5	6 000
	No. 1 to No. 0000.....	6	7 500
1001 to 2000.....	225 000 to 500 000 cir. mils.....	7	9 000
	525 000 to 1 000 000 cir. mils.....	8	10 000
	Over 1 000 000 cir. mils.....	9	11 000
	No. 14 to No. 8.....	5	6 000
	No. 7 to No. 2.....	6	7 500
2001 to 3000.....	No. 1 to No. 0000.....	7	9 000
	225 000 to 500 000 cir. mils.....	8	10 000
	525 000 to 1 000 000 cir. mils.....	9	11 000
	Over 1 000 000 cir. mils.....	10	12 500
	No. 10 to No. 8.....	7	9 000
3001 to 4000.....	No. 7 to No. 2.....	8	10 000
	No. 1 to No. 0000.....	8	10 000
	225 000 to 500 000 cir. mils.....	9	11 000
	525 000 to 1 000 000 cir. mils.....	10	12 500
	Over 1 000 000 cir. mils.....	11	13 500
4001 to 5000.....	No. 10 to No. 8.....	9	11 000
	No. 7 to No. 2.....	9	11 000
	No. 1 to No. 0000.....	9	11 000
	225 000 to 500 000 cir. mils.....	10	12 500
	525 000 to 1 000 000 cir. mils.....	11	13 500
5001 to 6000 (grounded).....	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	10	12 500
	225 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	10	12 500
5001 to 6000 (ungrounded).....	225 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	12	15 000
	225 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
6001 to 7000 (grounded).....	No. 8 to No. 0000.....	11	13 500
	225 000 to 1 000 000 cir. mils.....	11	13 500
	Over 1 000 000 cir. mils.....	12	15 000
	No. 8 to No. 0000.....	14	17 500
	225 000 to 1 000 000 cir. mils.....	14	17 500
6001 to 7000 (ungrounded).....	Over 1 000 000 cir. mils.....	15	19 000
	No. 8 to No. 0000.....	12	15 000
	225 000 to 1 000 000 cir. mils.....	12	15 000
	Over 1 000 000 cir. mils.....	13	16 500
	No. 8 to No. 0000.....	12	15 000
7001 to 8000 (grounded).....	225 000 to 1 000 000 cir. mils.....	12	15 000
	Over 1 000 000 cir. mils.....	13	16 500
	No. 8 to No. 0000.....	16	20 000
	225 000 to 1 000 000 cir. mils.....	16	20 000
	Over 1 000 000 cir. mils.....	17	21 000

<sup>a</sup> Building wire with  $\frac{3}{4}$ -in. wall is permitted in sizes 14 and 12 by the National Electrical Code and will be furnished unless otherwise specified.

<sup>b</sup> Sizes Nos. 16 and 18 are not recognized by the National Electrical Code for 600-v. service.

### High-Voltage Test

3. Each coil, reel, or length of wire or cable after vulcanization of the insulation shall successfully withstand, for a period of 5 min., the application of an alternating-current voltage of not less than that prescribed in Table II. Insulation less than  $\frac{3}{32}$  in. in thickness shall not be tested for electrical properties in water. At the option of the purchaser, a test of 500 v. may, however, be applied between the conductors and shield, or both, in the completed cable.

### Insulation Resistance

4. (a) Each coil, reel, or length of wire or cable after being subjected to the high-voltage test specified in Section 3, shall have an insulation resistance in megohms per 1000 ft. at a temperature of 60 F. (15.6 C.) of not less than the value of  $R$  as calculated from the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where:

$R$  = insulation resistance in megohms per 1000 ft.,

$K$  = constant of 2000,

$D$  = diameter over insulation, and

$d$  = diameter under insulation.

(b) When the temperature at which the insulation resistance is measured differs from 60 F. (15.6 C.), the resistance shall be converted to that at 60 F. (15.6 C.) by multiplying the measured value by a coefficient to be furnished by the manufacturer, corresponding to the temperature at which the measurement is made.

### Thickness

5. (a) The average thickness of the insulation shall be not less than that prescribed in Table II. The minimum

thickness shall be not less than 95 per cent of the thickness required by Table II for insulations less than  $\frac{1}{8}$  in. in thickness and not less than 90 per cent of the specified thickness for insulations of  $\frac{1}{8}$  in. and greater.

(b) The thicknesses prescribed in Table II apply to single-conductor cable and the individual conductors of multiple-conductor cables, leaded or braided, except special applications such as aerial, nonleaded submarine, and vertical riser cable.

NOTE.—In Appendix II of A.S.T.M. Specifications D 27 will be found an interpretation of the insulation thicknesses given in Table II.

(c) *Number of Measurements.*—When the lot of wire to be inspected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination of the thickness shall be made on each of two coils or reels taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of the thickness made on each coil or reel so selected.

(d) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of this character. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement shall be made after slicing

off the heavier side of the insulation.) In the case of multiple-conductor cable the measurements shall be made on the individual wires before being cabled.

#### Workmanship

6. (a) The insulation shall be homogeneous in character, tough, elastic and applied concentrically about the conductor and shall fit tightly thereto. Where the insulation is applied in more than one layer, adjacent layers shall be vulcanized into a homogeneous mass.

(b) *Shielding*.—Wire and cables not supported on insulators shall be shielded or suitably protected against corona in accordance with the detailed recommendations contained in Sections 20 to 24, inclusive, of A.S.T.M. Specifications D 27.

(c) *Covering*.—The insulation of braided wires up to No. 7 A.w.g., inclusive, shall be covered with a single cotton braid. On larger sizes the fibrous covering shall consist of a cable tape and cotton braid or two cotton braids. The cable tape may be applied before vulcanization. In multiple-conductor cables the individual conductors shall not be braid-covered unless specified.

(d) *Repairs or Joints*.—When repairs or joints are made in the insulation, the work shall be done in such manner that the repaired part of the joint, and all parts affected in the process, shall be as strong and durable electrically and mechanically as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 5.

#### Rejection

7. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness measurement on each of the remaining coils or reels shall be made.

(b) When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

(c) Each coil, reel, or length which fails to conform to the electrical requirements prescribed in these specifications shall be rejected.

#### Methods of Testing

8. All measurements and tests necessary for determining the conformity of the insulated wire and cable with these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470),<sup>3</sup> except as otherwise prescribed in these specifications.

# *Tentative Specifications for* INSULATED WIRE AND CABLE: POLYVINYL INSULATING COMPOUND<sup>1</sup>



A.S.T.M. Designation: D 734 - 43 T

ISSUED, 1943.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These specifications cover wire and cable insulated with a thermoplastic synthetic insulating compound made from polyvinyl chloride or its copolymer with vinyl acetate. The suitability of the insulation shall be determined solely by physical and electrical tests. This insulation is ordinarily recommended for use at conductor temperatures not in excess of 60 C. It is further ordinarily recommended that the minimum temperature at which the insulation shall be installed shall be not less than -10 C. (14 F). When the temperatures of operation and installation are in excess of these limits, and for voltages in excess of 600 v. the manufacturer shall be consulted for proper recommendations.

## Material

2. (a) The conductor shall be soft,

annealed copper and shall be uncoated unless otherwise specified. If a coating is specified, it shall conform to the Standard Specifications for Tinned Soft or Annealed Copper Wire for Electrical Purposes (A.S.T.M. Designation: B 33)<sup>3</sup> for tinned coating, or the Tentative Specifications for Lead-Coated and Lead-Alloy-Coated Soft Copper Wire for Electrical Purposes (A.S.T.M. Designation: B 189)<sup>3</sup> of the American Society for Testing Materials, for lead or lead alloy coating.

(b) Conductors of No. 6 A.w.g. and smaller shall be solid unless otherwise specified and shall conform to the Standard Specifications for Soft or Annealed Copper Wire (A.S.T.M. Designation: B 3) of the American Society for Testing Materials.<sup>3</sup>

## Strand

3. Each individual wire of a stranded conductor shall be considered separately and shall be designated as a strand.

## Shape

4. Each solid conductor and each

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1943.

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part I-B.

strand shall be round and reasonably free from imperfections.

### Stranding

5. The stranding shall conform to the Standard Specifications for Concentric-Lay-Stranded Copper Conductors, Hard, Medium-Hard, or Soft (A.S.T.M. Designation: B 8) Table II, class B or, where specified, class C, of the American Society for Testing Materials.<sup>3</sup>

### Permissible Variations in Diameter and Area

6. When the diameter of solid conductors and strands is specified, the permissible variation from the specified value shall not exceed 1 per cent under or 3 per cent over for wire 0.010 in. in diameter or larger, or 0.3 mil over for wire less than 0.010 in. in diameter. When the area of cross-section of stranded conductors is specified, the permissible variation shall not exceed 1 per cent under the specified value.

### Coating Test

7. *Retests and Rejections.*—If the specimens tested in accordance with Section 2 (a) fail to conform to requirements, two more specimens shall be tested. If one of these two additional specimens fail to meet requirements, that coil or reel shall be rejected. If both specimens are free from any signs of failure, the coil or reel shall be accepted. If more than 10 per cent of the samples in the entire order fail, all of the wire shall be rejected. If 10 per cent or less of the samples in the entire lot fail, each coil, reel, or length may be tested and accepted or rejected upon the results of the individual tests.

### Sampling and Methods of Test

8. All sampling, measurements, and tests necessary for determining con-

formity of the insulation to these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>4</sup> except as otherwise prescribed in these specifications.

### Test Specimens

9. (a) From each of the samples selected in accordance with the requirements of Section 3 of Methods D 470, test specimens shall be prepared as follows:

Initial Physical Properties	Number of Test Specimens
Tensile strength and ultimate elongation.....	3
Air oven test.....	3
Heat shock test.....	3
Heat distortion test.....	3
Flame test.....	3
Cold bend test.....	3
Oil resistance test.....	3

(b) One specimen from each set shall be used for the tests listed in Paragraph (a) and the remaining two specimens shall be held in reserve. If any of the specimens tested fails to conform to the values specified, that test shall be repeated on the two additional specimens from the same sample.

### Physical Properties

10. The insulation shall conform to the requirements as to physical properties prescribed in Table I.

TABLE I.—PHYSICAL TEST REQUIREMENTS FOR INSULATION.

Tensile strength, min., psi.....	1500
Elongation at rupture, min., per cent.....	100
Depreciation in tensile strength after 120 hr. in air oven test at 99 to 101 C., max., per cent.....	15
Depreciation in elongation after 120 hr. in air oven test at 99 to 101 C., max., per cent.....	40

### Heat Shock Test

11. The insulation shall not show any cracks after a single conductor insulated wire or cable has been wound around a

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

mandrel as specified in Table II and subjected to a temperature of 120 to 122 C. for 1 hr.

TABLE II.—MANDREL SIZES FOR HEAT SHOCK TEST.

Conductor Size, A.w.g. or Cir. Mils.	Number of Adjacent Turns	Mandrel Size, Multiply Outside Diameter of Insulated Wire by
No. 8 and under.....	6	1
No. 7 to No. 2, incl.....	6	2
No. 1.....	1	2
No. 0 to No. 0000.....	180-deg. bend	2
225 000 cir. mils. and over.....	180-deg. bend	5

### Heat Distortion Test

12. (a) The thickness of the insulation shall not decrease more than 50 per cent when subjected to a temperature of 120 to 122 C., under the load condition prescribed in Table III when tested

TABLE III.—GAGE LOADS FOR HEAT DISTORTION TESTS.

Conductor Size, A.w.g.	Load on Gage, g.
No. 18.....	300
No. 16.....	400
No. 15 to No. 8, incl.....	500
No. 7 to No. 1, incl.....	750
Nos. 0 to 0000, incl.....	1000
Buffed samples from conductors larger than No. 0000 A.w.g.....	2000

in accordance with the following Paragraphs (b) to (d).

#### (b) Sample Preparation:

(1) *Insulation on Conductors of No. 0000 A.w.g. and Smaller.*—Measure the initial diameter of a 1-in. specimen of the insulated conductor with a micrometer caliper with flat surface on each anvil. Also measure the diameter of the conductor, and calculate the thickness of the insulation as follows:

$$T_1 = \frac{D - C}{2}$$

where:

$T_1$  = original thickness of the insulation,

$D$  = initial diameter of the insulated conductor, and

$C$  = diameter of the conductor.

(2) *Insulation on Conductors Larger than No. 0000 A.w.g.*—Remove an 8-in. sample of insulation from the conductor and buff it to obtain parallel surfaces  $0.050 \pm 0.010$  in. in thickness. From this cut a test specimen 1 in. in length and  $\frac{9}{16} \pm \frac{1}{16}$  in. in width. Obtain the original thickness,  $T_1$ , of the specimen by using a gage<sup>5</sup> with a  $\frac{3}{8}$ -in. presser foot and no load on the foot of the gage.

(c) *Procedure.*—Adjust the gage<sup>5</sup> so that it would subject the specimen to be tested to the load specified in Table III if the specimen were in position in the gage, and then place the gage in a preheated oven for 1 hr. at a temperature of 120 to 122 C. At the end of this 1-hr. period place the test specimen in the oven. Allow the specimen and the gage to remain in the oven for 1 hr. at 120 to 122 C. At the end of the 1-hr. period place the specimen directly under the foot of the gage with the load specified in Table III applied to the specimen and allow to remain in the oven for a period of 1 hr. at 120 to 122 C. At the end of this 1-hr. period read the dial of the gage.

(d) *Calculation.*—In the case of insulated conductors of No. 0000 A.w.g. and smaller, calculate the thickness of the insulation after the heat distortion test as follows:

$$T_2 = \frac{F - C}{2}$$

where:

$T_2$  = thickness of the insulation after the heat distortion test,

$F$  = final diameter of insulated con-

<sup>5</sup> A Randall and Stickney gage, or equivalent, is satisfactory.



ductor as read from the gage (Paragraph (c)), and

$C$  = diameter of conductor.

In the case of insulated conductors larger than No. 0000 A.w.g., the thickness  $T_2$  of the insulation after the heat distortion test is numerically equal to the dial reading on the gage (Paragraph (c)).

### Flame-Retardant Properties

13. (a) The flame-retardant properties of the insulation shall be such that wire will not convey flame nor continue to burn for more than 1 min. after five 15-sec. applications of a standard test flame, the period between applications of such flame being 15 sec.

(b) The apparatus for this test shall consist of a test chamber of sheet metal 12 in. in width, 14 in. in depth, and 24 in. in height, open at the top, and provided with means of clamping the specimen at the upper end and supporting it in a vertical position; a means for adjusting the position of the specimen; a 4-lb. weight (for No. 8 A.w.g. and smaller sizes) to be attached to the lower end of the specimen to keep it taut; a Tirrill burner with an attached pilot light, mounted on a 20-deg.-angle block—the burner having a nominal bore of  $\frac{3}{8}$  in. and a length of approximately 4 in. above the primary air inlets; an adjustable steel angle (*jig*) attached to the bottom of the chamber to insure the correct location of the burner with relation to the sample; a supply of ordinary illuminating gas at normal pressure; a watch or clock with a hand which makes one complete revolution each minute; and flame indicators consisting of strips of gummed kraft paper, 5 mils in nominal thickness and  $\frac{1}{2}$  in. in width. The paper used for the indicators is what is known to the trade as 60-lb. stock and is substantially the same material as that described in Federal Specification for Tape; Paper, Gummed (Kraft) (UU-T-111a).

(c) The test shall be made in a room generally free from drafts of air, but a ventilated hood may be used if air currents do not affect the flame. One end of a test specimen of wire approximately 22 in. in length shall be clamped in position at the upper end of the chamber and, for No. 8 A.w.g. and smaller, the weight then attached to keep the specimen taut. A paper indicator shall be applied to the specimen so that the lower edge is 10 in. above the point at which the inner blue cone of the test flame is to be applied. The indicator shall be wrapped once around the specimen, with the gummed side toward the conductor and the ends pasted evenly together and projecting  $\frac{3}{4}$  in. from the wire on the opposite side of the specimen to that to which the flame is to be applied. The paper tab shall be moistened only to the extent that will permit proper adhesion. The height of the flame with the burner vertical shall be adjusted to 5 in., with an inner cone  $1\frac{1}{2}$  in. in height.

(d) The burner, with only the pilot lighted, shall be placed in front of the specimen so that the vertical plane through the stem of the burner includes the axis of the wire. The angle block shall rest against the jig, which shall be adjusted so that there is a distance of  $1\frac{1}{2}$  in. along the axis of the burner stem, between the tip of the stem and the surface of the specimen. The valve supplying the gas to the burner proper shall then be opened and the flame automatically applied to the specimen. This valve shall be held open for 15 sec. This process shall be repeated four times. During each application of the flame, the specimen shall be adjusted, if necessary, so that the tip of the inner blue cone touches the surface of the specimen. If more than 25 per cent of the extended portion of the indicator is burned after

the five applications of the flame, the wire shall be considered to have conveyed flame. The duration of the burning of the specimen after the fifth application of the flame shall be noted, and any specimen which continues to burn for more than 1 min. shall not be considered to be acceptable.

#### Oil Resistance Test

14. (a) The oil-resistant properties of the insulation shall be such that wire will conform to the requirements of the oil immersion test as described in the following Paragraphs (b) and (c).

(b) Test specimens of the insulated wire or cable shall be placed in S.A.E. 20 oil at a temperature of 69 to 71 C., so that the specimens are totally immersed except for the ends and shall remain in the oil for a period of 4 hr. At the end of this time, the specimens shall be removed from the oil, blotted to remove excess oil, and allowed to rest at room temperature for a period of 16 to 48 hr. The tensile strength and elongation of the insulation on these specimens shall then be determined at the same time as the original properties are determined.

(c) The depreciation in tensile strength and elongation after the 4 hr. oil test shall not exceed 15 per cent. There shall be no increase in the diameter of insulated wire after the 4-hr. oil test.

#### Cold Bend Test

15. (a) The insulation shall not show any cracks when a test specimen of the insulated wire has been subjected to the cold bend test as described in the following Paragraph (b).

(b) Test specimens of insulated wire shall be subjected to a temperature of -9 to -11 C. for a period of 1 hr. Upon removal from the cooling chamber, the specimens shall be wound immediately

around a mandrel as specified in Table IV. The winding or bending shall be done at an approximately uniform rate so that the time required is not more than 1 min.

TABLE IV.—MANDREL SIZES FOR COLD BEND TEST.

Conductor Size, A.w.g. Numbers or Cir. Mils.	Number of Adjacent Turns	Diameter of Mandrel, in.
No. 18.....	6	$\frac{5}{16}$
No. 16.....	6	$\frac{3}{8}$
No. 14 <sup>a</sup> .....	6	$\frac{1}{2}$
No. 12 <sup>a</sup> .....	6	$\frac{5}{8}$
No. 10 <sup>a</sup> .....	6	$\frac{3}{4}$
No. 8 <sup>a</sup> .....	6	$\frac{11}{16}$
No. 6.....	6	$1\frac{1}{4}$
No. 4.....	6	$1\frac{3}{8}$
No. 2.....	6	$1\frac{1}{2}$
No. 1.....	6	$2\frac{1}{16}$
No. 0.....	6	$2\frac{7}{8}$
No. 00.....	6	3
No. 000.....	6	$3\frac{1}{4}$
No. 0000.....	6	$3\frac{3}{4}$
250 000 to 500 000 cir. mils.....	180-deg. bend	8 times cable diameter
Over 500 000 cir. mils.	180-deg. bend	10 times cable diameter

<sup>a</sup> On sizes Nos. 14, 12, and 10 A.w.g. having insulation thicknesses greater than  $\frac{3}{4}$  in., and on size No. 8 A.w.g. having insulation thicknesses greater than  $\frac{1}{2}$  in., the mandrel diameters shall be  $\frac{3}{4}$  in.,  $\frac{1}{2}$  in.,  $\frac{3}{4}$  in., and  $\frac{1}{2}$  in., respectively.

#### High Voltage Test

16. Each coil, reel or length of single conductor insulated wire or cable shall be immersed in water and tested with the alternating current voltage as prescribed in Table V.

#### Insulation Resistance

17. (a) Each coil, reel or length of single conductor wire or cable after being subjected to the high-voltage test as specified in Section 16 shall have an insulation resistance not less than that prescribed in Paragraph (b).

(b) The insulated conductor, No. 14 A.w.g. or larger, shall have an insulation resistance at 60 F. (15.6 C.) of not less than that given by the following formula:

$$R = K \log_{10} \frac{D}{d}$$

where:

$R$  = insulation resistance in megohms per 1000 ft.,

$K$  = constant = 500 at 60 F. (15.6 C.),

$D$  = diameter over insulation, and

$d$  = diameter over conductor.

(c) In calculating insulation resistance the temperature coefficients given in Table VI shall be used.

### Thickness of Insulation

18. (a) The average thickness of the insulation shall be not less than prescribed in Table V. The minimum thickness shall be not less than 90 per cent of the thickness prescribed in Table V.

(b) When the lot of wire to be in-

spected consists of two coils or reels, or less, at least one determination of the thickness shall be made on each coil or reel. When the lot consists of more than two coils or reels and less than 20 coils or reels, at least one determination shall be made on each of two coils taken at random. If the lot consists of 20 or more coils or reels, not less than 10 per cent of the coils or reels shall be selected at random and at least one determination of thickness made on each coil or reel so selected.

(c) *Measurement of Thickness.*—The thickness measurements may be made with any type of micrometer reading to 0.001 in., suitable for measurements of

TABLE V.—THICKNESS OF INSULATION AND A-C. TEST VOLTAGES.

NOTE 1.—Thicknesses in Column A are recommended for use on wire and cables installed in appliances and apparatus in recognized metal raceways, or where used for supervisory control systems, etc.

NOTE 2.—Thicknesses in Column B are recommended for station control wires and cables or similar important circuits or other installations where a heavier wall than specified in Column A is desired.

NOTE 3.—Thicknesses in Column C are recommended for non-lead-covered cable installed in wet locations. If cable has lead or other impervious sheath, the thicknesses in Column B should be used. Underground ducts and direct earth burial shall be considered wet locations.

NOTE 4.—When the provisions of the National Electric Code have jurisdiction, the wire or cable shall comply with the Underwriters' Laboratories Standard for Synthetic-Insulated Wires and Cables, dated October, 1942.

NOTE 5.—For applications in excess of 600 v., the manufacturers' recommendations shall be obtained.

Size of Conductor, A.w.g. numbers or cir. mils	Column A Insulation Thickness, sixty-fourths of an inch	A-C. Test Voltage	Column B Insulation Thickness, sixty-fourths of an inch	A-C. Test Voltage	Column C Insulation Thickness, sixty-fourths of an inch	A-C. Test Voltage
No. 18 to No. 16.....	2	1000	3	1500	4	2000
No. 14 to No. 9.....	2	1500	3	2000	4	2000
No. 8.....	3	1500	4	2000	5	3500
No. 7 to No. 2.....	4	2000	4	2000	5	3500
No. 1 to No. 0000.....	5	2500	5	2500	6	4000
225 000 to 500 000.....	6	3000	6	3000	7	4500
525 000 to 1 000 000.....	7	3500	7	3500	8	6000
Over 1 000 000.....	8	4000	8	4000	9	8000

TABLE VI.—TEMPERATURE COEFFICIENTS.

Temperature		Temperature Coefficient
Fahrenheit Deg.	Centigrade Deg.	
50	10.0.....	0.29
53	11.7.....	0.45
56	13.3.....	0.66 <sup>a</sup>
59	15.0.....	0.91
60	15.6.....	1.00
62	16.7.....	1.26
65	18.3.....	1.80
68	20.0.....	2.50
71	21.7.....	3.50
74	23.3.....	5.00
77	25.0.....	6.60
80	26.7.....	8.30
83	28.3.....	11.0
86	30.0.....	17.5

<sup>a</sup> Editorially corrected, October, 1946.

this character. The average thickness of the insulation shall be taken as one-half the difference between the mean of the maximum and the minimum diameters measured at any point and the average diameter of the conductor measured at the same point. The minimum thickness shall be taken as the difference between a measurement made over the conductor plus the thinnest wall and the diameter of the conductor. (The first measurement shall be made by slicing off the heavier side of the insulation.)

In the case of multiple-conductor cable, the measurements shall be made on the individual wires before being cabled.

#### Workmanship

19. (a) The insulation shall be homogeneous in character, tough, flexible and applied concentrically about the conductor and shall fit tightly thereto.

(b) *Repairs and Joints.*—When repairs or joints are made in the insulation, the work shall be done in such a manner that the repaired part of the joint, and all parts affected by the process, shall meet the same electrical tests as the remainder of the insulation and shall not exceed the limitations on the thickness specified in Section 18.

#### Rejection

20. (a) If the thickness of the insulation of any coil or reel is found to be less than the specified value, that coil or reel shall be rejected and a thickness

measurement on each of the remaining coils or reels shall be made.

(b) When 10 or more samples are selected from any inspection lot, all coils, reels, or lengths may be rejected if more than 10 per cent of the samples fail to conform to the requirements as to physical properties. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. Where the number of samples selected in any inspection lot is less than 10, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

(c) Each coil, reel, or length which fails to conform to the electrical requirements of these specifications shall be rejected.

# *Tentative Specifications for*

## RUBBER SHEATH COMPOUND FOR ELECTRICAL INSULATED CORDS AND CABLES<sup>1</sup>



**A.S.T.M. Designation: D 532 - 46 T**

ISSUED, 1939; REVISED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover a durable, vulcanized rubber compound suitable for use as the outside covering or sheath on insulated electrical cords and cables.

### Character of Sheath

2. The vulcanized rubber sheath shall be homogeneous in character, tough and elastic.

### Methods of Testing

3. All measurements and tests necessary for determining the conformity of the sheath to these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Administrative Committee on Standards, September 9, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Physical Requirements

4. The vulcanized rubber sheath shall conform to the following requirements as to physical properties:

Tensile strength, min., psi.....	3500
Tensile stress at 200 per cent elongation, min., psi.....	500
Elongation at rupture, min., per cent....	500
Set in 2-in. gage length, max., in.....	$\frac{1}{2}$
Tensile strength after 96 hr. in oxygen bomb test, min., psi.....	2500
Elongation at rupture after 96 hr. in oxygen bomb test, min., per cent.....	400
Tear resistance, min., lb. per in.....	40

### Sampling for Original Physical Tests

5. (a) Cord or cable having an outside diameter up to and including 1 in. shall be sampled in accordance with the following requirements:

	Number of Samples
2 000 ft. and under.....	none
2 001 to 10 000 ft.....	1 for each 2000 ft. or fraction thereof
10 001 to 100 000 ft.....	5 plus 1 for each 10,000 ft. additional over 10 000 ft.
100 001 to 500 000 ft.....	14 plus 1 for each 25 000 ft. over 100 000 ft.

(b) For cable larger than 1 in. in outside diameter, one sample shall be selected from quantities between

1000 and 2000 ft. and for larger quantities double those specified in Paragraph (a).

#### Sampling for Accelerated Aging Tests

6. (a) No samples shall be selected where the quantity is less than 2000 ft. of cord or cable.

(b) For cord or cable having an outside diameter up to and including 1 in., one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. and one additional sample for each additional 25,000 ft. thereafter.

(c) For cable larger than 1 in. in outside diameter one sample shall be selected for each quantity ordered between 2000 ft. and 10,000 ft. of cable and one additional sample for each additional 10,000 ft. thereafter.

#### Test Specimens

7. (a) For the physical tests and accelerated aging tests the test specimen

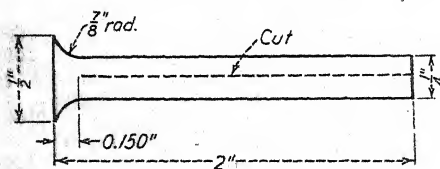


FIG. 1.—Test Specimen for Tear Test.

shall be taken from the completed cord or cable and cut parallel to the axis of the cord or cable. With the exception of the tear test, the test specimen shall be either a segment or sector cut with a sharp knife or a shaped specimen cut out with a die and shall have an area of cross-section not greater than 0.025 sq. in., after irregularities, corrugations, and reinforcing cords or wires are removed by buffing.

(b) The test specimen for the tear test shall be cut with a sharp knife or die. After irregularities, corrugations, and reinforcing cords or wires are removed the test specimen shall conform

to the dimensions shown in Fig. 1. The thickness in inches of the test specimen shall not be greater than 0.150 in. and not less than 0.040 in. and shall be split longitudinally with a new razor blade to a point 0.150 in. from the wider end.

#### Tear Test

8. (a) The tear test shall be made on a minimum of six individual test specimens prepared as described in Section 7 (b). The two halves of the split end of the test specimen shall be placed in the jaws of the tension testing machine and the jaws separated at the rate of 20 in. per min. The tear resistance shall be determined by dividing the load in pounds required to tear the section by the thickness of the test specimen in inches. The average of the values obtained on all test specimens shall conform to the requirement for tear resistance prescribed in Section 4.

(b) If the average tear resistance of the first set of test specimens is less than the requirement prescribed in Section 4, two other sets of specimens shall be tested and if either of these sets fails, the coils, reels, or lengths shall be rejected.

#### Rejection

9. When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. When the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

# *Tentative Specifications for*

## GR-M POLYCHLOROPRENE SHEATH COMPOUND FOR ELECTRICAL INSULATED CORDS AND CABLES<sup>1</sup>



A.S.T.M. Designation: D 752 - 46 T

ISSUED 1943; REVISED, 1944, 1946.\*

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover a durable vulcanized GR-M polychloroprene compound suitable for use as the outside covering or sheath on insulated electrical cords and cables for heavy duty service at temperatures not lower than  $-25^{\circ}\text{C}$ .

### Character of Sheath

2. The vulcanized polychloroprene sheath shall be homogeneous in character, tough and elastic, and shall be properly vulcanized to conform to the requirements prescribed in these specifications.

### Methods of Testing

3. All measurements and tests necessary for determining the conformity of the sheath to these specifications shall be made in accordance with the Tentative

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.

Prior to their publication as tentative, these specifications were issued as an emergency standard (ES-28) from April 15, 1943, to December 27, 1943.

Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications.

### Physical Requirements

4. The vulcanized polychloroprene sheath shall conform to the following requirements as to physical properties:

Tensile strength, min., psi.....	1800
Tensile stress at 200 per cent elongation, min., psi.....	500
Elongation at rupture, min., per cent.....	300
Set in 2-in. gage length, max., in.....	$\frac{1}{8}$
Tensile strength after 96 hr. in oxygen bomb test, min., psi.....	1600
Elongation at rupture after 96 hr. in oxygen bomb test, min., per cent.....	250
Tensile strength after 168 hr. in air oven test at 157 to 159 F. ( $69.4$ to $70.6^{\circ}\text{C}$ ), min., psi.....	1600
Elongation at rupture after 168 hr. in air oven test at 157 to 159 F. ( $69.4$ to $70.6^{\circ}\text{C}$ ), min., per cent.....	250
Tensile strength and elongation at rupture after 18 hr. in oil immersion test at 249 to 251 F. ( $120.6$ to $121.7^{\circ}\text{C}$ ), min., per cent of the original value.....	60

### Sampling for Original Physical Tests

5. For physical tests, samples of cord and cable shall be selected as follows:

\* Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



(a) Cord or cable having an outside diameter up to and including 1 in. shall be sampled in accordance with the following requirements:

	Number of Samples
2000 ft. and under.....	none
2001 to 10 000 ft.....	1 for each 2000 ft. or fraction thereof
10 001 to 100 000 ft.....	5 plus 1 for each 10 000 ft. additional over 10 000 ft.
100 001 to 500 000 ft.....	14 plus 1 for each 25 000 ft. over 100 000 ft.

(b) For cable larger than 1 in. in outside diameter, one sample shall be selected from quantities between 1000 and 2000 ft. and for larger quantities double those specified in Paragraph (a).

#### Sampling for Accelerated Aging and Oil Immersion Tests

6. For accelerated aging and oil immersion tests, samples of cord and cable shall be selected as follows:

(a) No samples shall be selected where the quantity is less than 2000 ft. of cord or cable.

(b) For cord or cable having an outside diameter up to and including 1 in., one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. and one additional sample for each additional 25,000 ft. thereafter.

(c) For cable larger than 1 in. in outside diameter, one sample shall be selected for each quantity ordered between 2000 ft. and 10,000 ft. of cable and one additional sample for each 10,000 ft. thereafter.

#### Test Specimens

7. For the physical, accelerated aging, and oil immersion tests, the test specimens shall be taken from the completed cord or cable and cut parallel to the axis of the cord or cable. The test specimen shall be either a segment or a sector cut with a sharp knife, or a shaped specimen cut out with a die and shall have an area of cross-section not greater

than 0.025 sq. in. after irregularities, corrugations, and reinforcing cords or wires are removed by buffing.

#### Oil Immersion Test

8. (a) Specimens obtained in accordance with Section 7 after being measured and prepared for testing shall be completely immersed in oil conforming to the requirements prescribed in Paragraph (c) at a temperature of 249 to 251 F. (120.6 to 121.7 C.) for a period of 18 hr. At the end of the required immersion time, the specimens shall be removed from the oil, blotted lightly to remove excess oil, suspended in air at room temperature for  $4 \pm \frac{1}{2}$  hr., after which they shall be tested for tensile strength and elongation.

(b) The calculations for tensile strength shall be based on the cross-sectional area of the specimen obtained before immersion in the oil. Likewise, the elongation shall be based on the gage marks applied to the specimen before immersion in the oil.

(c) The general characteristics of the oil shall conform to the following requirements:

	Requirement	A.S.T.M. Designation <sup>a</sup>
A.S.T.M. color number, max.....	3	D 155
Condition.....	clear	D 97
Flash point, min.....	235 C. (454 F.)	D 92
Fire point, min.....	280 C. (536 F.)	D 92
Free sulfur, max., per cent.....	none	....
Neutralization number, max., mg. of KOH per g.....	0.020	D 663
Pour point, max.....	-5 C. (+23 F.)	D 97
Specific gravity at 60 F., min.....	0.93	D 287
Total sulfur, max., per cent.....	0.35	D 129
Viscosity, sec.: at 30 C. (86 F.) (Saybolt Furol).....	550 to 650	D 88
at 100 C. (212 F.) (Saybolt Universal).....	95 to 105	D 88

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials:

Tentative Method of Test for Color of Lubricating Oil and Petroleum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155),<sup>4</sup>

Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92),<sup>4</sup>

Tentative Method of Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration (A.S.T.M. Designation: D 663),<sup>4</sup>

Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97),<sup>4</sup>

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287),<sup>4</sup>

Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (A.S.T.M. Designation: D 129),<sup>4</sup> and

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>4</sup>

### Rejection

9. When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil,

reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. When the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

## *Tentative Specifications for*

### GR-M POLYCHLOROPRENE SHEATH COMPOUND FOR ELECTRICAL INSULATED CORDS AND CABLES WHERE EXTREME ABRASION RESISTANCE IS NOT REQUIRED<sup>1</sup>



A.S.T.M. Designation: D 753 - 44 T

ISSUED, 1943; REVISED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. These specifications cover a durable, vulcanized, GR-M polychloroprene compound suitable for use as the outside covering or sheath on insulated electrical cords and cables where extreme abrasion resistance is not required at temperatures not lower than  $-25^{\circ}\text{C}$ .

#### Character of Sheath

2. The vulcanized polychloroprene sheath shall be homogeneous in character, tough and elastic, and shall be properly vulcanized to conform to the requirements prescribed in these specifications.

#### Methods of Testing

3. All measurements and tests necessary for determining the conformity of the sheath to these specifications shall

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1944.

Prior to their publication as tentative, these specifications were issued as an emergency standard (ES-30) from April 15, 1943, to December 27, 1943.

be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications.

#### Physical Requirements

4. The vulcanized polychloroprene sheath shall conform to the following requirements as to physical properties:

Tensile strength, min., psi.....	1200
Elongation at rupture, min., per cent.....	250
Set in 2-in. gage length, max., in.....	$\frac{1}{8}$
Tensile strength after 96 hr. in oxygen bomb test, min., psi.....	1000
Elongation at rupture after 96 hr. in oxygen bomb test, min., per cent.....	200
Tensile strength after 168 hr. in air oven test at 157 to 159 F. (69.4 to 70.6 C.), min., psi.....	1000
Elongation at rupture after 168 hr. in air oven test at 157 to 159 F. (69.4 to 70.6 C.), min., per cent.....	200
Depreciation in tensile strength and elongation at rupture after 18 hr. in oil immersion test at 249 to 251 F. (120.6 to 121.7 C.), max., per cent.....	40

#### Sampling for Original Physical Tests

5. For physical tests, samples of cord and cable shall be selected as follows:

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(a) Cord or cable having an outside diameter up to and including 1 in. shall be sampled in accordance with the following requirements:

	Number of Samples
2000 ft. and under.....	none
2001 to 10 000 ft.....	1 for each 2000 ft. or fraction thereof
10 001 to 100 000 ft.....	5 plus 1 for each 10 000 ft. additional over 10 000 ft.
100 001 to 500 000 ft.....	14 plus 1 for each 25 000 ft. over 100 000 ft.

(b) For cable larger than 1 in. in outside diameter, one sample shall be selected from quantities between 1000 and 2000 ft., and for larger quantities double those specified in Paragraph (a).

#### Sampling for Accelerated Aging and Oil Immersion Tests

6. For accelerated aging and oil immersion tests, samples of cord and cable shall be selected as follows:

(a) No samples shall be selected where the quantity is less than 2000 ft.

(b) For cord or cable having an outside diameter up to and including 1 in., one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. and one additional sample for each additional 25,000 ft. thereafter.

(c) For cable larger than 1 in. in outside diameter one sample shall be selected for each quantity ordered between 2000 ft. and 10,000 ft. of cable and one additional sample for each additional 10,000 ft. thereafter.

#### Test Specimens

7. For the physical, accelerated aging, and oil immersion tests the test specimens shall be taken from the completed cord or cable and cut parallel to the axis of the cord or cable. The test specimen shall be either a segment or sector cut with a sharp knife, or a shaped specimen cut out with a die and shall have an area of cross-section not greater

than 0.024 sq. in. after irregularities, corrugations, and reinforcing cords or wires are removed by buffing.

#### Oil Immersion Test

8. (a) Specimens obtained in accordance with Section 7 after being measured and prepared for testing shall be completely immersed in oil conforming to the requirements prescribed in Paragraph (c) at a temperature of 249 to 251 F. (120.6 to 121.7 C.) for a period of 18 hr. At the end of the required immersion time, the specimens shall be removed from the oil, blotted lightly to remove excess oil, suspended in air at room temperature for  $4 \pm \frac{1}{2}$  hr., after which they shall be tested for tensile strength and elongation.

(b) The calculations for tensile strength shall be based on the cross-sectional area of the specimen obtained before immersion in the oil. Likewise, the elongation shall be based on the gage marks applied to the specimen before immersion in the oil.

(c) The general characteristics of the oil shall conform to the following requirements:

	Requirement	A.S.T.M. Designation <sup>a</sup>
A.S.T.M. color number, max....	3	D 155
Condition.....	clear	....
Flash point, min.....	235 C. (454 F.)	D 92
Fire point, min.....	280 C. (536 F.)	D 92
Free sulfur, max., per cent.....	none	....
Neutralization number, max., mg. of KOH per g.....	0.020	D 663
Pour point, max.....	-5 C. (+23 F.)	D 97
Specific gravity at 60 F., min....	0.93	D 287
Total sulfur, max., per cent....	0.35	D 129
Viscosity, sec.: at 30 C. (86 F.) (Saybolt Furol).....	550 to 650	D 88
at 100 C. (212 F.) (Saybolt Universal).....	95 to 105	D 88

<sup>a</sup> These designations refer to the following methods of the American Society for Testing Materials: Tentative Method of Test for Color of Lubricating Oil and Petroleum by Means of A.S.T.M. Union Colorimeter (A.S.T.M. Designation: D 155).<sup>4</sup>

Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>4</sup>

Tentative Method of Test for Acid and Base Numbers of Petroleum Oils by Color-Indicator Titration (A.S.T.M. Designation: D 663).<sup>4</sup>

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

Standard Method of Test for Cloud and Pour Points (A.S.T.M. Designation: D 97),<sup>4</sup>

Standard Method of Test for Gravity of Petroleum and Petroleum Products by Means of the Hydrometer (A.S.T.M. Designation: D 287),<sup>4</sup>

Standard Method of Test for Sulfur in Petroleum Oils by Bomb Method (A.S.T.M. Designation: D 129),<sup>4</sup> and

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88).<sup>4</sup>

### Rejection

9. When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail. If

10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. When the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected, if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

# *Tentative Specifications for*

## GR-S SYNTHETIC RUBBER SHEATH COMPOUND FOR ELECTRICAL INSULATED CORDS AND CABLES<sup>1</sup>



A.S.T.M. Designation: D 866 - 46 T

ISSUED, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover a durable, vulcanized synthetic rubber compound, type GR-S, suitable for use as the outside covering or sheath on insulated electrical cords and cables. Recommended use of this compound in service requiring flexing is limited to temperatures of  $-25^{\circ}\text{C}$ . and higher (Note).

NOTE.—GR-S jackets suitable for operation at lower temperatures can be provided when agreed upon by the purchaser and the seller.

### Character of Sheath

2. The vulcanized synthetic rubber sheath shall be homogeneous in character, tough and elastic, and shall be properly vulcanized to conform to the requirements prescribed in these specifications.

### Methods of Testing

3. All measurements and tests necessary for determining the conformity of the sheath to these specifications shall be made in accordance with the Tentative

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-like Materials.

<sup>2</sup> Accepted by the Administrative Committee on Standards, January 10, 1946.

Prior to their publication as tentative, these specifications were issued as Emergency Specifications ES-6 from May 8, 1942, to January 10, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation: D 470) of the American Society for Testing Materials,<sup>3</sup> except as otherwise prescribed in these specifications.

### Physical Requirements

4. The vulcanized synthetic rubber sheath shall conform to the following requirements as to physical properties:

Tensile strength, min., psi.....	1800
Elongation at rupture, min., per cent....	300
Set in 2-in. gage length, max., in.....	$\frac{3}{8}$
Tensile strength after 48 hr. in oxygen pressure aging test, min., psi.....	1400
Elongation after 48 hr. in oxygen pressure aging test, min., per cent.....	200

### Sampling for Original Physical Tests

5. (a) Cord or cable having an outside diameter up to and including 1 in. shall be sampled in accordance with the following requirements:

	Number of Samples
2000 ft. and under.....	none
2001 to 10 000 ft.....	1 for each 2000 ft. or fraction thereof
10 001 to 100 000 ft.....	5 plus 1 for each 10 000 ft. additional over 10 000 ft.
100 001 to 500 000 ft.....	14 plus 1 for each 25 000 ft. over 100 000 ft.

(b) For cable larger than 1 in. in outside diameter, one sample shall be selected from quantities between 1000 and 2000 ft., and for larger quantities double those specified in Paragraph (a).

#### Sampling for Accelerated Aging Tests

6. (a) No samples shall be selected where the quantity is less than 2000 ft. of cord or cable.

(b) For cord or cable having an outside diameter up to and including 1 in., one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. and one additional sample for each additional 25,000 ft. thereafter.

(c) For cable larger than 1 in. in outside diameter one sample shall be selected for each quantity ordered between 2000 ft. and 10,000 ft. of cable and one additional sample for each additional 10,000 ft. thereafter.

#### Test Specimens

7. For the physical tests and accelerated aging tests the test specimen shall be taken from the completed cord or

cable and cut parallel to the axis of the cord or cable. The test specimen shall be either a segment or sector cut with a sharp knife or a shaped specimen cut out with a die and shall have an area of cross-section not greater than 0.024 sq. in., after irregularities, corrugations, and reinforcing cords or wires are removed by buffing.

#### Rejection

8. When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. When the number of samples selected in any inspection lot is less than ten, all coils, reels, or lengths shall be rejected, if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.



# *Tentative Specifications for*

## GR-S SYNTHETIC RUBBER SHEATH COMPOUND FOR ELECTRICAL INSULATED CORDS AND CABLES WHERE EXTREME ABRASION RESISTANCE IS NOT REQUIRED<sup>1</sup>



A.S.T.M. Designation: D 812 - 44 T

ISSUED, 1944.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These specifications cover a vulcanized synthetic rubber compound, type GR-S, suitable for use as the outside covering, or sheath, on insulated electrical cords and cables which are not subject to severe mechanical use and where extreme abrasion resistance is not required.

### Character of Sheath

2. The vulcanized sheath shall be homogeneous in character, tough and elastic, and shall be properly vulcanized to conform to the requirements prescribed in these specifications.

### Methods of Testing

3. All measurements and tests necessary for determining the conformity of the sheath to these specifications shall be made in accordance with the Tentative Methods of Testing Rubber Insulated Wire and Cable (A.S.T.M. Designation:

D 470),<sup>3</sup> except as otherwise prescribed in these specifications.

### Physical Requirements

4. The vulcanized sheath shall conform to the following requirements as to physical properties:

Tensile strength, min., psi.....	1200
Elongation at rupture, min., per cent.....	250
Tensile strength after 48 hr. in 70 C., oxygen bomb test, min., psi.....	1000

NOTE.—Where these jackets are to be exposed to sunlight it is suggested a sunlight resisting wax be incorporated in the compound.

### Sampling for Original Physical Tests

5. (a) Cord or cable having an outside diameter up to and including 1 in. shall be sampled in accordance with the following requirements:

	Number of Samples
2000 ft. and under.....	none
2001 to 10 000 ft.....	1 for each 2000 ft. or fraction thereof
10 001 to 100 000 ft.....	5 plus 1 for each 10 000 ft. additional over 10 000 ft.
100 001 to 500 000 ft.....	14 plus 1 for each 25 000 ft. over 100 000 ft.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Accepted by Committee E-10 on Standards, December 13, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) For cable larger than 1 in. in outside diameter, one sample shall be selected from quantities between 1000 and 2000 ft., and for larger quantities double those specified in Paragraph (a).

#### Sampling for Accelerated Aging Tests

6. (a) No samples shall be selected where the quantity is less than 2000 ft. of cord or cable.

(b) For cord or cable having an outside diameter up to and including 1 in., one sample shall be selected for each quantity ordered between 2000 ft. and 25,000 ft. and one additional sample for each additional 25,000 ft. thereafter.

(c) For cable larger than 1 in. in outside diameter one sample shall be selected for each quantity ordered between 2000 ft. and 10,000 ft. of cable and one additional sample for each additional 10,000 ft. thereafter.

#### Test Specimens

7. For the physical tests and accelerated aging tests the test specimen shall be taken from the completed cord

or cable and cut parallel to the axis of the cord or cable. The test specimen shall be either a segment or sector cut with a sharp knife or a shaped specimen cut out with a die and shall have an area of cross-section not greater than 0.024 sq. in., after irregularities, corrugations, and reinforcing cords or wires are removed by buffing.

#### Rejection

8. When ten or more samples are selected in any inspection lot, all coils, reels, or lengths shall be rejected if more than 10 per cent of the samples fail. If 10 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests. When the number of samples selected in any inspection lot is less than 10, all coils, reels, or lengths shall be rejected, if more than 20 per cent of the samples fail. If 20 per cent or less fail, each coil, reel, or length may be tested and shall be accepted or rejected upon the results of such individual tests.

# Tentative Methods of TESTING RUBBER INSULATED WIRE AND CABLE<sup>1</sup>



A.S.T.M. Designation: D 470 - 46 T

ISSUED, 1946; REVISED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. These methods describe procedures for the testing of rubber-insulated wire and cable. They are not to be considered completely applicable to all types of wire and cable, nor do they necessarily include every test applicable to a particular type. To determine the tests to be made on a particular wire or cable, reference should be made to the specifications for that type. These methods do not apply to the class of products known as flexible cords. The insulation resistance and moisture absorption tests do not apply to the class of products having a separator between the conductor and the insulation.

## PHYSICAL TESTS OF INSULATION

### Physical Tests

2. Physical tests include the determination of tensile strength, tensile stress, ultimate elongation, permanent set, and accelerated aging.

<sup>1</sup> Under the standardization procedure of the Society these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Reverted to tentative by action of the Society at annual meeting, June, 1946; revision accepted by the Administrative Committee on Standards, September 9, 1946.

Prior to their present publication as tentative, these methods were published as tentative from 1937 to 1941, being revised in 1938, 1939, and 1940. They were adopted in 1941, published as standard from 1941 to 1946, when they were revised and republished as tentative.

## Sampling

3. (a) *Number of Samples.*—Wire and cable shall be sampled as follows:

For sizes less than 250,000 cir. mils, one sample shall be selected for each quantity ordered between 2000 ft. and 50,000 ft. of wire or cable and one additional sample for each additional 50,000 ft. thereafter. No sample shall be selected from lots of less than 2000 ft.

For sizes 250,000 cir. mils and over, one sample shall be selected for each quantity ordered between 1000 ft. and 25,000 ft. of wire or cable and one additional sample for each additional 25,000 ft. thereafter. No sample shall be selected from lots of less than 1000 ft.

(b) *Size of Samples.*—Samples shall be at least 6 ft. in length when the wire size is less than 250,000 cir. mils, and at least 3 ft. in length when the wire size is 250,000 cir. mils or over.

## Test Specimens

4. (a) *Number of Specimens.*—From each of the samples selected in accordance with Section 3, test specimens shall be prepared as follows:

	Number of Test Specimens
For determination of initial properties (unaged):	
Tensile strength, tensile stress, and ultimate elongation . . . . .	3
Permanent set . . . . .	3

## For aging tests:

Air pressure heat or oxygen pressure.	3
Air oven.....	3

One specimen of each three shall be tested and the other two specimens held in reserve except that when only one sample is selected all three specimens shall be tested and the average of the results reported.

(b) *Size of Specimens.*—In the case of wire and cable smaller than No. 6 A.w.g. having an insulation thickness less than  $\frac{3}{8}$  in., the test specimen may be the entire section of the insulation. When the full cross-section is used, the specimens shall not be cut longitudinally. In the case of wire and cable of No. 6 A.w.g. and larger, or in the case of wire and cable smaller than No. 6 A.w.g. having an insulation thickness greater than  $\frac{3}{8}$  in., specimens approximately square in section, with a cross-section not greater than 0.025 sq. in., shall be cut from the insulation. In extreme cases, it may be necessary to use a segmented or sector-shaped specimen. The test specimens shall be approximately 6 in. in length.

(c) *Preparation of Specimens.*—The test specimen shall have no surface incisions and be as free as possible from other imperfections. Surface irregularities, such as corrugations due to stranding, etc., shall be removed by buffing. When a test specimen is buffed, care shall be taken to make it smooth and of uniform thickness.

NOTE.—The removal of the rubber insulation can be greatly accelerated, and in most cases a test specimen which is an entire section can be obtained free from surface incisions and imperfections, by means of mercury. The mercury should be introduced at one end of the sample between the insulation and the tinned surface of the conductor, and the sample inclined on a support with the end to which the mercury is applied at the top. The separation of the rubber insulation results from the amalgamation of the tin of the conductor with the mercury. The amalgamation is assisted by first immersing and rubbing the tinning on the exposed end of the conductor in the mercury.

(d) *Condition and Age.*—Samples of the insulated wire or cable for physical and accelerated aging tests shall be taken after vulcanization and prior to the application of any covering except that a cable tape may be applied before vulcanizing. No tests shall be made within 24 hr. nor later than 60 days after vulcanization unless agreed to by the manufacturer. Specimens shall not be heated, immersed in water, or subjected to any mechanical or chemical treatment not specifically prescribed in these methods. Specimens for accelerated aging tests having cable tape applied prior to vulcanization shall be aged with such tape removed.

## Calculation of Area of Specimens

5. Calculation of the area of a test specimen shall be made as follows:

(a) Where the total cross-section of the insulation is used, the area shall be taken as the difference between the area of the circle whose diameter is the average outside diameter of the insulation and the area of the conductor. The area of a stranded conductor shall be calculated from its maximum diameter.

(b) Where a slice cut from the insulation by a knife held tangent to the wire is used, and the slice so cut has the cross-section of a segment of a circle, the area shall be calculated as that of the segment of a circle whose diameter is that of the insulation. The height of the segment of the wall of insulation on the side from which the slice is taken. (The values may be obtained from a table giving the areas of segments of a unit circle for the ratio of the height of the segment to the diameter of the circle.)

(c) Where the cross-section of the slice is not a segment of a circle, the area shall be calculated from a direct measurement of the volume or from the specific gravity and the weight of a known length of the specimen having a uniform cross-section.

(d) Where a portion of a sector of a circle has to be taken when the conductor is large and the insulation thin, the area shall be calculated as the thickness times the width. (This applies either to a straight test specimen or one stamped out with a die, and assumes that corrugations have been removed by buffing.)

(e) Where a portion of a sector of a circle has to be taken when the conductor is large and the insulation thick, the area shall be calculated as the proportional part of the area of the total cross-section.

(f) The dimensions of specimens to be aged shall be determined before the aging cycle is begun.

### Physical Test Procedures

6. (a) The physical properties shall be determined in accordance with the Standard Methods of Tension Testing of Vulcanized Rubber (A.S.T.M. Designation: D 412) of the American Society for Testing Materials,<sup>3</sup> except as specified in the following Paragraphs (b) to (e).

(b) The specimens to be tested shall be at a temperature of 20 to 28 C.

(c) For all physical tests, specimens marked with gage marks 2 in. apart shall be placed in the jaws of the testing machine with a maximum distance between jaws of 4 in.

(d) Tensile stress shall be determined as that load per unit area which produces the elongation of the sample prescribed by the specifications.

(e) The set test shall be made on a separate test specimen having a length of not less than 6 in. and marked with gage marks 2 in. apart. The specimen shall be placed in the jaws of the testing machine with a maximum distance between jaws of 4 in. and shall be stretched at the rate of 20 in. per min. (jaw speed) until the gage marks are 6 in. apart. The test specimen shall then be released

within 5 sec. and the distance between gage marks shall be determined 1 min. after the beginning of release. The set is the difference between this length and the original 2-in. gage length.

### Aging Test Procedure

7. (a) Specimens shall be aged in accordance with the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (A.S.T.M. Designation: D 572)<sup>3</sup> or the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573),<sup>3</sup> and the Standard Method of Air Pressure Heat Test of Vulcanized Rubber (A.S.T.M. Designation: D 454)<sup>3</sup> of the American Society for Testing Materials, except as specified in the following Paragraphs (b) and (c).

(b) The period of aging shall be as prescribed in the specifications.

(c) In not less than 16 nor more than 48 hr. after the completion of the aging process, the aged specimens shall be subjected to tensile strength and ultimate elongation tests in accordance with Section 6. Physical tests on both aged and unaged specimens shall be made at the same time.

### Retests

8. If any specimen fails to conform to the values which may be specified for any test, either before or after aging, that test shall be repeated on two additional specimens from the same sample.

### Report

9. The report shall include the following:

(a) Calculated values of tensile strength, tensile stress, ultimate elongation, and set,

(b) All observed and recorded data on which the calculations are based,

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

- (c) Date of vulcanization of the rubber, if known,
- (d) Dates of all tests,
- (e) Ambient temperatures during the period of physical testing,
- (f) Type of testing machine used,
- (g) Method of aging, and
- (h) Time of aging.

## ELECTRICAL TESTS OF INSULATION

### *Voltage Tests*

#### Types of Voltage Tests

10. Voltage tests shall include the following:

A. *Voltage Test on Entire Cable*

B. *Double Voltage Test on Short Samples*

C. *Cold-Bend, Long-Time Voltage Test on Short Samples*

#### Apparatus

11. (a) Voltage tests shall be made with alternating current, except when direct current is specified.

(b) For tests with a-c. voltage, there shall be used a transformer of such capacity that the crest factor (ratio of maximum to mean effective) of the test voltage shall not differ by more than plus or minus 10 per cent from that of a sinusoidal wave when the transformer is loaded with the test specimen. Voltage shall be measured by a tertiary coil and voltmeter or by an electrostatic voltmeter or potential transformer with suitable low-voltage indicator, connected to the secondary, or high-voltage side of the test transformer. The frequency of the test voltage shall be nominally between 25 and 60 cycles per second, inclusive.

(c) For tests with d-c. voltage there shall be used a battery, generator or suitable rectifying equipment supplied with alternating current. Voltage shall be measured with an electrostatic voltmeter, or in the case of the rectifying equipment, with suitable low-voltage

indicators, provided the latter are so connected that their indications are independent of the test load. The test rectifier shall at all times be so loaded, if necessary by the use of additional capacitive ballast, that the d-c. ripple does not exceed 4 per cent.

#### Application of Voltage

12. (a) *Single Conductor Cables.*—Single conductor cables shall be tested between conductor and metallic sheath or water in which they are immersed.

(b) *Multiple Conductor Cables.*—In the case of multiple conductor cables, each conductor shall be tested against adjacent conductors and against the sheath or water.

#### Rate of Voltage Application

13. Voltage shall be increased from zero to the prescribed test value at a uniform rate of rise approximating but not exceeding 3 kv. per sec.

#### A. *Voltage Test on Entire Cable:*

##### Procedure

14. Wire and cable shall be subjected to a-c. voltage as specified. When d-c. is specified, the d-c. voltage shall be 2.2 times the required a-c. voltage. The duration of test shall be 5 min. Tests shall be made as follows:

(a) *General.*—Where insulated conductors are subjected to a high voltage test in water they shall be immersed in a grounded water bath and tested while still immersed.

(b) *Single Conductor.*—Single conductors shall be tested after 12-hr. immersion and without covering except a tape or braid applied before vulcanization. Rubber sheathed conductors shall be immersed and tested after the sheath is applied. Where a metallic sheath is applied, the conductors shall be retested against the sheath.

(c) *Multiple Conductor.*—The indi-

vidual conductors of multiple conductor cables shall be tested before assembling as prescribed in Paragraph (b). After assembly, each conductor shall be tested against the adjacent conductors. Where a metallic sheath is applied, each conductor adjacent to the sheath shall, in addition, be tested against the sheath. Cables having a rubber or other non-metallic waterproof sheath shall be immersed for 12 hr. and the conductors tested against ground. Cables covered with cable tape and braid or double braid with a weather-resistant finish shall not be immersed for testing. Cables having a metallic armor shall be tested after the application of the armor at a voltage equal to 80 per cent of the test voltage before armoring. Such cables shall be immersed for 12 hr. prior to test only if for submarine use and if no lead sheath or shielding is applied.

#### *B. Double- Voltage Test on Short Samples:*

##### **Sampling**

15. One sample, 10 ft. or more in length as may be required, shall be taken from each 10,000 ft. or fraction thereof of each type of cable, with a minimum of three samples. Tests upon lots of less than 10,000 ft. shall be made only when specifically requested in the purchase order. Samples may be taken either from process or from completed cable, at the option of the manufacturer.

##### **Procedure**

16. At least one-half of the total length of each test specimen shall be in contact with the grounded terminal during the period of test. This part of each specimen shall be immersed in water at room temperature, after the removal of any metallic coverings, for a period of 1 hr. prior to testing. At the end of this time, each test specimen shall be subjected for 5 min. to a voltage equal to twice that specified in Section 14.

#### *C. Cold-Bend, Long-Time Voltage Test on Short Samples:*

##### **Sampling**

17. One 10-ft. sample shall be taken from each 10,000 ft. or fraction thereof of completed cable. Tests upon lots of less than 10,000 ft. shall be made only when specifically requested in the purchase order.

##### **Procedure**

18. (a) Each sample shall be subjected to a temperature of  $-10^{\circ}\text{C}$ . for a period of 2 hr. Immediately at the end of this period, the sample shall be bent 180 deg. around a cylindrical mandrel and then straightened. It shall then be bent 180 deg. around the mandrel in the opposite direction. The cable shall be so held during bending operations that it cannot revolve around its own axis.

(b) The diameter of the mandrel shall be as follows:

Thickness of Conductor In- sulation, Sixty- fourths of an Inch	Mandrel Diameter as a Multiple of Over-all Cable Diameter	
	Up to 500,000 cir. mils	500,000 cir. mils and Over
10 to 12 . . . .	8	10
13 to 20 . . . .	10	12
21 and over . .	12	12

(c) Immediately following the bending test, the cable, while still bent, shall be subjected to the test voltage specified in Section 14 for a period of 2 hr.

##### **Report**

19. The report shall include the following:

- Type of test,
- Time and place of test,
- Test voltages,
- Duration of each test, and
- Result of each test, including location of any failure.



*Insulation Resistance Tests<sup>4</sup>***Apparatus**

20. The apparatus shall consist of the following:

(a) *Source of Potential.*—A source of constant potential of from 100 to 500 v., and

(b) *Galvanometer and Resistances.*—A galvanometer with suitable shunts, and a calibrating resistance of at least 0.1 megohm. The galvanometer should have a high current sensitivity. A sensitivity of the order of  $10^{-9}$  amp. per cm. with scale at a meter distance is desirable. The most convenient shunt is the type known as a universal shunt whereby the current through the galvanometer may be changed by powers of ten without changing its damping.

**Procedure**

21. (a) Single conductor cables shall be tested between conductors and sheath or water in which they are immersed. Tests on multiple conductor cables with unshielded conductors shall be made between each conductor and all other conductors and the sheath or water. Tests on multiple conductor cables with shielded conductors shall be made between each conductor and shield. The conductor of the wire or cable shall be connected to the negative terminal, and the sheath and other conductors, if any, to the positive terminal of the apparatus. On short sections of wire or cable, a guard circuit should be used to prevent end leakage.

(b) The deflection of the galvanometer caused by passing current through the calibrating resistance shall be first be determined. The wire or cable shall then be connected into the circuit. At the end of 1 min. after closing the circuit, the galvanometer deflection shall again be noted, and the temperature of the wire or cable observed.

**Calculations**

22. Insulation resistance is commonly expressed in terms of a standard length. The insulation resistance,  $R$ , of a standard length may be determined as follows:

$$R = M \times \frac{D}{d} \times \frac{S}{s} \times \frac{l}{l_s}$$

where:

$M$  = calibrating resistance,

$D$  = deflection produced with calibrating resistance in circuit,

$S$  = shunt ratio with calibrating resistance in circuit,

$d$  = deflection produced with test specimen in circuit,

$s$  = shunt ratio with test specimen in circuit,

$l$  = length of entire wire or cable, and

$l_s$  = standard length.

**Temperature Correction**

23. The insulation resistance of wires and cables varies widely with temperature. If the temperature at which the measurement was made differs from 60 F. (15.6 C), the resistance shall be reduced to that at 60 F. (15.6 C.) by multiplying the measured value by the temperature coefficient specified for the insulation in question.

*Moisture Absorption Test<sup>4</sup>***Procedure**

24. A 15-ft. sample of the insulated wire or cable shall be taken after vulcanization and prior to the application of any coverings except tape applied before vulcanizing. Such cable tape shall be removed before making the moisture absorption test. The middle 10 ft. of the sample shall be immersed in distilled water for a period of 14 days with the 2.5-ft. portion at each end kept above the

<sup>4</sup> See Section 1.

water as leakage insulation. The specific inductive capacity of the insulation shall be determined after 1, 7, and 14 days with the water at the same temperature for each measurement. The water shall be maintained at room temperature but not less than 70 F. (21 C.).

#### Calculations

25. The specific inductive capacity shall be determined at commercial frequencies or 1000 cycles and shall be calculated as follows:

$$\text{Specific inductive capacity} = 13,600 C \log_{10} \frac{D}{d}$$

where:

$C$  = capacity in microfarads of 10 ft. of sample,

$D$  = diameter over the insulation, and  
 $d$  = diameter over the conductor.

The moisture absorption test shall be made only when requested by the purchaser.

#### OZONE RESISTANCE TEST

##### Purpose

26. The purpose of this method is to test the resistance of rubber insulation to ozone attack which may be encountered in connection with the operation of high-voltage cable.

##### Special Solutions Required

27. (a) *Starch Indicator Solution*.—Stir 1 g. of soluble of starch into 40 ml. of cold water, heat to boiling, while stirring constantly, until starch is thoroughly dissolved, dilute with cold water to about 200 ml., and add 2 g. of crystallized zinc chloride. Let solution settle for some time and pour off for use the supernatant liquid. Renew every 2 or 3 days. A fresh solution of 1 g. of soluble starch in 100 ml. of boiling water may also be used. When using these starch solutions as an indicator, add a few drops of acetic acid

(10 per cent) to the solution being titrated.

(b) *Standard Iodine Solution*.—Place in a weighing tube 2 g. of KI and 10 ml. of water, and weigh the tube and solution. Add iodine directly to the solution in the weighing tube on the balance pan until the total iodine in solution is about 0.1 g. Accurately weigh the solution with the added iodine. Determine the amount of iodine added to the solution. Remove the weighing tube, pour the solution into a beaker, wash the weighing tube held over the beaker with distilled water, pour the solution from the beaker into a volumetric flask, rinse the beaker into a 1000-ml. volumetric flask with distilled water, and dilute the solution in the flask to 1 liter. This solution is fairly stable if kept in a cool, dark place in a well-stoppered, brown bottle.

(c) *Sodium Thiosulfate Solution*.—Prepare a  $\text{Na}_2\text{S}_2\text{O}_3$  solution of approximately the same strength as the standard iodine solution by placing about 0.24 g. of  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  in a 1000-ml. volumetric flask and dilute to 1 liter. Since this solution gradually loses its strength, standardize it against the standard iodine solution on the day tests are run. The method for calculating the strength of the  $\text{Na}_2\text{S}_2\text{O}_3$  solution is described in Section 32 (a).

(d) *Potassium Iodide Solution*.—Dissolve about 20 g. of pure KI in 2 liters of water.

(e) *Acetic Acid (10 per cent)*.

##### Apparatus

28. (a) The apparatus shall consist of:

(1) A device for generating a controlled amount of ozone,

(2) A means for circulating ozonized air under controlled conditions of humidity and temperature through a chamber containing the specimens to be tested, and

(3) A means for determining the percentage of ozone concentration.

(b) A convenient form of apparatus is shown in Fig. 1. Air flows from an air pump, or compressed air supply through a series of chambers as indicated. The acid drier consists of two 500-ml. gas washing bottles filled to 30 per cent of capacity with  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84). In series with this is a second drier containing anhydrous calcium chloride or anhydrous calcium sulfate, and a U-tube hygrometer containing a small quantity of anhydrous copper sulfate which is used as an indicator of moisture. The rate of air flow is indicated by a calibrated flow meter. Ozonization of the air is accomplished in a generator consisting of a pair of concentric electrodes,

the inspection of specimens, without opening the chamber, during test. Near the bottom of the test chamber is placed a filter consisting of a layer of loose mineral wool held between two perforated grills. The ozonized air is led from the generator to the space below this filter. Temperature control may be secured by keeping apparatus in an air-conditioned room or by partially immersing the chamber in a water bath, which may be connected to a hot and cold water system, or provided with electric heaters thermostatically controlled. A thermometer is inserted into the cover of the test chamber, with its bulb as near the test specimens as prac-

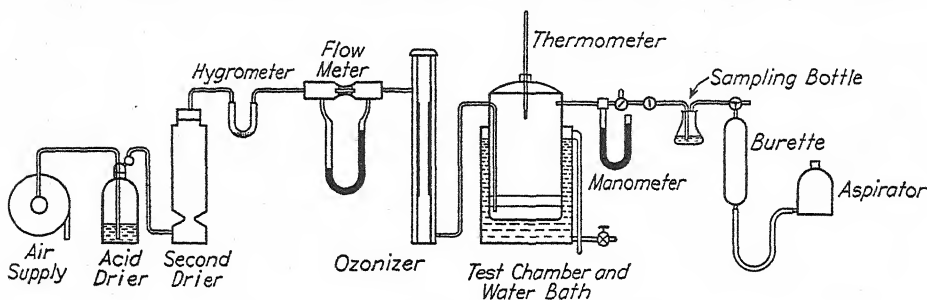


FIG. 1.—Apparatus for Ozone Resistance Test.

NOTE.—If the acid in the first acid drier should carry over into the second acid drier, a scrubber bottle may be inserted between the two scrubber bottles.

separated by a thin glass dielectric, between which voltage may be applied. This generator may be conveniently supplied by a potential transformer, equipped with variable voltage control, of 20 to 30-kv. rating. The test chamber should be of a material not attacked by ozone, and should be just large enough to accommodate the largest samples which it may be desired to test. Excessive size results in greater delay in obtaining the desired ozone concentration. A chamber 18 to 20 in. in length, and having a capacity of 2000 to 5000 cu. in., will generally be required. A convenient form of chamber is a glass jar, having a full-size cover. This permits easy access to the interior, and also

ticable. Discharge from the test chamber is accomplished by a double cock arrangement, one acting as a direct discharge into the outside atmosphere and the other, when normally closed, acting as a by-pass testcock. A manometer is inserted in the outlet pipe to indicate pressure in the test chamber. For determining ozone concentration a sampling bottle is connected to the outlet. Connection is made from this through a two-way stopcock to the top of a 500-ml. gas collecting burette. The bottom of this burette is connected by rubber tubing to a 500-ml. aspirator bottle. The use of rubber tubing should be avoided for those connections carrying ozonized air.

## Test Specimens

29. (a) Two specimens for the ozone test shall be selected beyond a point not less than 5 ft. from the end of the reel or coil to be tested. Where protective coverings are applied, such coverings shall be removed when nonadherent to insulation. However, where tapes or sheaths are applied directly to the insulation prior to vulcanization and left in place during vulcanization and are consequently adherent to insulation in the completed cable, such coverings shall not be removed.

(b) Each specimen shall be examined to make sure it is free from mechanical defects, such as cuts, dents, tears, loose threads, etc. One specimen shall be bent in the direction and plane of the existing curvature of the cable around a mandrel and through the specified angle in accordance with Paragraphs (c) and (d). The other specimen shall be bent similarly but in the reverse direction.

(c) Specimens less than 1 in. in diameter shall be bent, without twisting at room temperature, but not less than 20 C., around a mandrel the diameter of which is not less than three and one-half nor more than four and one-half times the maximum diameter of the cable in the unbent form. Specimens 1 in. and larger in maximum diameter shall be bent around a mandrel the diameter of which is not less than five and one-half nor more than six and one-half times the maximum diameter of the cable.

(d) The specimen shall be bent around a brass, aluminum, or suitably treated wooden mandrel of the specified diameter, as shown in Fig. 2, binding it with twine or tape where the ends cross. If the cable is too rigid to permit crossing of the ends it may be bent in the form of a U and tied so that at least an 180-deg. bend of the proper diameter is obtained.

(e) The surface of the insulation on

each specimen shall be wiped with a clean cloth to remove dirt or sweat. The bent specimens on their mandrels shall then be placed in a desiccator for 30 to 45 min. after bending to remove surface moisture and until placed in the ozone chamber.

(f) The specimens on their mandrels shall be placed in the ozone chamber which has been in equilibrium operation for at least 45 min. prior to inserting the

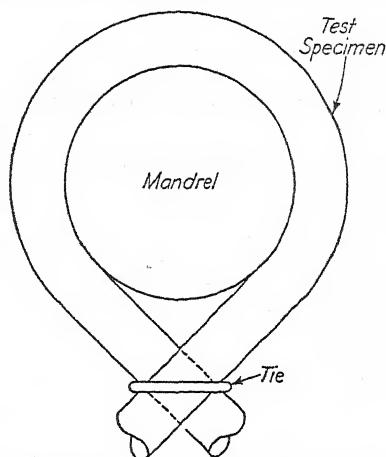


FIG. 2.—Specimen Bent Around Mandrel for Ozone Resistance Test.

specimens. Care shall be taken not to handle the insulation. The specimens shall be supported in an approximately vertical plane midway between the floor and ceiling with the free ends down to, but not touching, the floor.

(g) After the required exposure, the specimens shall be taken out of the chamber, all coverings removed from the specimens, and the specimens examined by the unaided eye for cracks in the bent portion. Cracks occurring in the 180-deg. sector including the tie, that is, where the specimen is not curved to conform to the mandrel, shall not be considered failures.

## Procedure

30. The air shall be circulated through the test apparatus at a constant rate of

flow for at least 15 min. prior to bending of the specimens. The flow shall be between 10 and 20 cu. ft. per hr. as indicated on the flow meter. The manometer shall indicate a slight pressure above atmospheric in the test chamber. This pressure may be controlled by the degree of closure of the discharge cock. After the ozone has been generated for a few minutes, a check shall be made on the percentage of ozone concentration (Section 31). The voltage of the ozone generator shall then be regulated so as to give a concentration of ozone of not less than 0.010 nor more than 0.015 per cent by volume. The temperature of the air in the test chamber shall be regulated to  $25 \pm 2$  C. When constant test conditions are obtained, the specimens shall be inserted in the test chamber and allowed to remain for a period of 1 hr.

#### Determination of Ozone Concentration

31. (a) *Collection of Sample.*—A 100-ml. portion of KI solution (1 per cent), slightly acidulated by a few drops of acetic acid, shall be placed in the sampling bottle and the latter connected to the sampling cock and gas burette as shown in Fig. 1. The two-way stopcock on the burette shall then be opened to the air and the burette filled to the mark with water by lifting the aspirator bottle. The stopcock shall be closed to the air and opened to the sampling bottle, and the sampling cock on the test chamber shall be opened. The aspirator bottle shall be lowered until the burette is emptied. When this point is reached, 500 ml. of gas will have bubbled through the KI solution. The stopcocks shall then be closed and the bottle withdrawn for titration.

(b) *Analysis of Sample.*—The solution in the bottle shall have a few drops of freshly prepared starch solution added

as an indicator and shall then be titrated with the standardized  $\text{Na}_2\text{S}_2\text{O}_3$  solution.

#### Calculations

32. (a) *Sodium Thiosulfate Solution.*—The strength of the  $\text{Na}_2\text{S}_2\text{O}_3$  solution shall be calculated as follows:

$$E = \frac{F \times C}{S}$$

where:

- $E$  = iodine equivalence of  $\text{Na}_2\text{S}_2\text{O}_3$  expressed as milligrams of iodine per milliliter of  $\text{Na}_2\text{S}_2\text{O}_3$ ,
- $F$  = number of milliliters of the iodine solution,
- $C$  = concentration of iodine in milligrams per milliliter, and
- $S$  = number of milliliters of  $\text{Na}_2\text{S}_2\text{O}_3$  used to titrate the solution.

(b) *Ozone.*—Since 1 mg. of iodine is equivalent to 0.1 ml. of ozone at room temperature and pressure (within the accuracy of this method of analysis at average room temperature and pressure), the ozone may be calculated as follows:

$$O = E \times 0.1$$

where:

- $O$  = number of milliliters of ozone at room temperature and pressure equivalent to 1 ml. of  $\text{Na}_2\text{S}_2\text{O}_3$  solution used, and
- $E$  = iodine equivalent of  $\text{Na}_2\text{S}_2\text{O}_3$  expressed as milligrams of iodine per milliliter of  $\text{Na}_2\text{S}_2\text{O}_3$ .

then:

$$\text{Percentage of ozone} = \frac{S \times O}{M} \times 100$$

where:

- $S$  = number of milliliters of  $\text{Na}_2\text{S}_2\text{O}_3$  used to titrate the solution,
- $O$  = number of milliliters of ozone at room temperature and

pressure equivalent to 1 ml. of  $\text{Na}_2\text{S}_2\text{O}_3$  solution used, and

$M$  = number of milliliters of the sample collected.

## TESTS ON FINISHED WIRE AND CABLE

### *Standard Horizontal Flame Test*

#### Apparatus

33. The apparatus shall consist of the following:

(a) *Test Chamber*.—A test chamber of sheet metal 12 in. in width, 14 in. in depth, and 24 in. in height which is open at the top and front and provided with means for supporting the test specimen in a horizontal position.

(b) *Tirrill Burner*.—A Tirrill burner having a bore of  $\frac{3}{8}$  in. and a length of 4 in. above the primary air inlets.

(c) *Watch*.—A watch or clock having a hand that makes one complete revolution per minute.

(d) *Gas*.—A supply of ordinary illuminating gas at normal pressure.

#### Procedure

34. The test shall be made in a room generally free from drafts of air, but a ventilated hood may be used if air currents do not affect the flame. A test specimen 10 in. in length shall be centered in a horizontal position on supports 8 in. apart. The height of the flame, with the burner vertical, shall be adjusted to 5 in., with an inner blue cone  $1\frac{1}{2}$  in. in height. The burner in a vertical position shall then be brought up to the specimen so that the inner blue cone just touches the underside of the specimen at a point midway between the supports. In this position the flame shall be directed against the specimen for a period of 30 sec. and then removed. During the test, as well as after the application of the flame, observation shall be made to determine whether or

not the area of the specimen supporting the flame extends outside the area exposed to the flame. The behavior and duration of the flaming of the specimen after the application of the test flame shall also be noted.

### *Vertical Flame Test*

#### Apparatus

35. The apparatus shall consist of the following:

(a) A test chamber of sheet metal 12 in. in width, 14 in. in depth, and 24 in. in height, open at the top, and provided with means for clamping the sample at the upper end and supporting it in a vertical position.

(b) Means for adjusting the position of the sample.

(c) A 4-lb. weight (for No. 8 A.w.g. and smaller sizes of wire) to be attached to the lower end of the sample to keep it taut.

(d) A Tirrill burner with an attached pilot light, mounted on a 20-deg. angle block. The burner shall have a nominal bore of  $\frac{3}{8}$  in., and a length of approximately 4 in. above the primary air inlets.

(e) An adjustable steel angle (jig) attached to the bottom of the chamber to insure the correct location of the burner with relation to the sample.

(f) A supply of ordinary illuminating gas at normal pressure.

(g) A watch or clock with a hand which makes one complete revolution each minute.

(h) Flame indicators consisting of strips of gummed Kraft paper, 5 mils in nominal thickness and  $\frac{1}{2}$  in. in width. The paper used for the indicators is that known to the trade as 60-lb. stock, and is material substantially the same as that described in Federal Specification for Tape; Paper, Gummed (Kraft) (UU-T-111a).

## Procedure

36. (a) The test shall be made in a room generally free from drafts of air, but a ventilated hood may be used if air currents do not affect the flame. One end of a test specimen of wire approximately 22 in. in length shall be clamped in position at the upper end of the chamber, and the weight then attached to keep the specimen taut. A paper indicator shall be applied to the specimen so that the lower edge is 10 in. above the point at which the inner blue cone of the test flame is to be applied. The indicator shall be wrapped once around the specimen, with the gummed side toward the conductor and the ends pasted evenly together and projecting  $\frac{3}{4}$  in. from the wire on the opposite side of the specimen to that to which the flame is to be applied. The gummed surface of the paper tab shall be moistened only to the extent that will permit proper adhesion. The height of the flame with the burner vertical shall be adjusted to 5 in., with an inner cone  $1\frac{1}{2}$  in. in height, by means of the main supply valve.

(b) The burner, with only the pilot lighted, shall be placed in front of the specimen so that the vertical plane through the stem of the burner includes the axis of the wire. The angle block shall rest against the jig, which shall be adjusted so that there is a distance of  $1\frac{1}{2}$  in., along the axis of the burner stem, between the tip of the stem and the surface of the specimen. The valve supplying the gas to the burner proper shall then be opened and the flame automatically applied to the specimen. This valve shall be held open for 15 sec. and then closed for 15 sec., and this process repeated four times. During each application of the flame, the specimen shall be adjusted, if necessary, so that the tip of the inner blue cone touches the surface of the specimen.

If more than 25 per cent of the extended portion of the indicator is burned after the five applications of the flame, the wire shall be considered to have conveyed flame.

## Moisture Absorption Test on Fibrous Coverings

### Apparatus

37. The apparatus shall consist of a desiccator containing anhydrous calcium chloride, a set of mandrels having diameters as indicated in Table I,

TABLE I.—DIAMETER OF MANDREL FOR BENDING SPECIMENS IN MOISTURE TEST.

Size of Wire, A.w.g. numbers or circular mils	Diameter of Mandrel, in.	Size of Wire, A.w.g. numbers or circular mils	Diameter of Mandrel, in.
No. 14	$\frac{3}{32}$ "	450 000 cir. mils	$\frac{69}{64}$ "
No. 12	$\frac{9}{64}$ "	500 000 cir. mils	$\frac{69}{64}$ "
No. 10	$\frac{5}{16}$ "	550 000 cir. mils	$10\frac{1}{2}$ "
No. 8	$\frac{3}{8}$ "	600 000 cir. mils	11
No. 6	$1\frac{1}{4}$ "	650 000 cir. mils	$11\frac{1}{4}$ "
No. 4	$1\frac{3}{8}$ "	700 000 cir. mils	$11\frac{1}{2}$ "
No. 2	$1\frac{9}{16}$ "	750 000 cir. mils	12
No. 1	$2\frac{1}{16}$ "	800 000 cir. mils	$12\frac{1}{4}$ "
No. 0	$2\frac{7}{16}$ "	850 000 cir. mils	$12\frac{1}{2}$ "
No. 00	$3$ "	900 000 cir. mils	$12\frac{3}{4}$ "
No. 000	$3\frac{1}{4}$ "	950 000 cir. mils	$13\frac{1}{4}$ "
No. 0000	$3\frac{1}{2}$ "	1 000 000 cir. mils	$13\frac{3}{4}$ "
250 000 cir. mils	$5\frac{19}{16}$ "	1 250 000 cir. mils	$17\frac{3}{4}$ "
300 000 cir. mils	$5\frac{1}{2}$ "	1 500 000 cir. mils	$18\frac{1}{2}$ "
350 000 cir. mils	$5\frac{7}{8}$ "	1 750 000 cir. mils	$19\frac{1}{4}$ "
400 000 cir. mils	$6\frac{1}{4}$ "	2 000 000 cir. mils	$20\frac{3}{4}$ "

\* The values in the table apply throughout to conductors having two fibrous coverings. For Nos. 14, 12, 10, and 8 A.w.g. conductors having one fibrous covering, the mandrel diameters shall be  $\frac{1}{16}$ ,  $\frac{3}{16}$ ,  $\frac{1}{8}$ , and  $\frac{1}{4}$  in., respectively.

a quick-damping balance accurate to 10 mg., and an agitated, constant-temperature bath of distilled water which can be maintained at a temperature of  $21 \pm 0.5$  C. The bath shall be either fitted with a cover to keep out dust, or placed within a tight enclosure during the test. If, at any time, the water becomes dirty, or shows the presence of a surface film of dust or wax, it shall be replaced by fresh water.

### Procedure

38. (a) Before cutting a test specimen to size, the coil or other wire to be tested



shall be permitted to attain a room temperature of not less than 21 C. Handling and flexing of wire samples to be tested shall be reduced to the absolute minimum necessary in testing.

(b) A  $24 \pm \frac{1}{4}$ -in. specimen of wire shall be cut from the coil or other sample of wire, and bent around a mandrel of the diameter indicated in Table I. For No. 2 A.w.g. or smaller wires, as many turns of the test specimen shall be made about the mandrel as will permit it to conform closely to the mandrel, with a 2 to  $2\frac{1}{2}$ -in. straight length of the specimen at each end. Adjacent turns shall not touch each other, but shall be separated about  $\frac{1}{8}$  to  $\frac{1}{4}$  in. For wire sizes larger than No. 2 A.w.g., a simple U-turn of the specimen shall be made about the mandrel.

(c) The specimen shall be removed from the mandrel without disturbing its form, and placed in the desiccator over anhydrous calcium chloride at a room temperature of not less than 21 C. for at least 18 hr. It shall then be removed from the desiccator, weighed to the nearest 10 mg., and the weight recorded as  $W$ .

(d) The specimen shall then be immersed in the distilled water bath, with  $1 \pm \frac{1}{8}$  in. of each end of the coil or U-bend projecting above the surface of the water. After 24 hr. of immersion, the specimen shall be removed from the bath, shaken vigorously for 5 sec. to remove adherent moisture, weighed again, and the weight recorded as  $W_1$ . The weighing shall be completed within 2 min. after removal from the bath. All fibrous coverings other than tape shall then be removed from the full length of

the specimen, and the conductor, insulation, and tape, if any, shall be weighed and the weight recorded as  $W_2$ .

(e) The moisture absorption of the specimen shall be expressed as a percentage of the moisture absorbed by the fibrous covering, and shall not be corrected for the portion of the wire projecting above the water. The percentage of absorption may be calculated (to 0.1 per cent) by means of the formula:

Moisture absorption, per cent =

$$\frac{100 \times (W_1 - W)}{W - W_2}$$

#### *Mineral Filler Content Determination*

##### **Procedure**

39. (a) The mineral filler content shall be determined in the following manner. The outer braid together with adhering compound shall be removed from a 6-in. section of finished wire or cable. This shall be weighed and the weight recorded as  $A$ . The weighed sample shall then be ashed and the weight of ash recorded as  $B$ . All weights shall be determined to the nearest 0.01 g. The percentage of mineral filler is obtained by dividing  $B$  by  $A$  and multiplying by 100.

(b) In construction involving two or more over-all braid coverings, the mineral filler content of the complete braid covering shall be determined by the method specified above for the outer braid. A 6-in. section of the completed braid covering from the finished wire or cable, together with all adhering compound, shall be used for the test.

# *Tentative Specifications for*

## CELLULAR RUBBER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 798 - 46a T

(Revision of Specifications D 798 - 46 T)

ISSUED, 1944; REVISED, 1944, JANUARY 1946, JUNE 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These specifications cover cellular rubber products of two types, grouped into four classifications and include sponge rubbers, latex foam rubbers, and expanded rubbers. The base material used in their manufacture may be natural rubber, reclaimed rubber, synthetic rubber, or rubber-like materials, alone or in combination. Ebonite cellular rubbers are not included in these specifications.

(b) In case of conflict between the provisions of these general specifications and those of detailed specifications or methods of test for a particular product the latter shall take precedence.

### Description of Terms

2. (a) *Cellular Rubbers*.—Cellular rubber products all contain cells which are defined as small hollow receptacles. The cells may either be open and inter-

connecting or closed and not interconnecting.

(b) *Rubber*.—The term rubber is used to include both natural and the synthetic types.

(c) *Skin*.—The smooth surface of the cellular rubber product, formed by contact with the mold or cover plates, is defined as a natural skin. On some products it is desirable to add a solid rubber skin coating. This may be accomplished by applying a solid rubber coating to the uncured rubber and vulcanizing them together, or it may be applied to the vulcanized cellular rubber product by dipping or otherwise coating. The use to which the cellular rubber product is to be put determines the thickness of added skin required. Products subject to abrasion or those which must withstand absorption of water or gases will ordinarily require an added skin coating. In all cases there should be good adhesion between the added skin and the cellular rubber itself.

### Manufacture

3. (a) *Sponge Rubbers*.—Sponge rubbers are made by incorporating into the compound an inflating agent, such as

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

By publication of these specifications, the American Society for Testing Materials does not undertake to insure anyone utilizing the specifications against liability for infringement of Letters Patent nor assume any such liability, and such publication should not be construed as a recommendation of any patented or proprietary application that may be involved.

<sup>2</sup> Latest revisions accepted by the Administrative Committee on Standards, January 10, 1946, and by the Society at annual meeting, June, 1946.

sodium bicarbonate, that gives off a gas which expands the mass during the vulcanization process. Sponge rubbers are made from slab or solid rubbers and are manufactured in sheet, strip, molded, or special shapes. Unless otherwise specified, sheet and strip sponge rubber

ture of latex foam rubbers consists of a network of open or interconnecting cells. Latex foam rubbers are made from rubber latices or liquid rubbers. They are manufactured in sheet, strip, molded, or special shapes. Latex foam rubbers shall have a vulcanized cellular structure

TABLE I.—PHYSICAL REQUIREMENTS OF CELLULAR RUBBERS TYPE R, CLASS RN, NON-OIL RESISTANT.

(See Section 10 for Methods of Testing)

Basic Requirements			Requirements Added by Suffix Letter				
Grade Number	Compression Deflection, 25 per cent Deflection (Limits), psi.	Indentation, 25 per cent Deflection (Limits), psi. <sup>a</sup>	Suffix A	Suffix B	Suffix F	Suffix H	Suffix L
			Oven Aged 7 days at 158 F., Change from Original Load-Deflection or Indentation Values (Limits), per cent	Compression-Set, 70 hr. at 158 F., 50 per cent Deflection, max., per cent	Change from Original Deflection, max., per cent	Flexing Test, Compression-Set, max., per cent	Water Absorption, max., per cent
SPONGE RUBBERS							
RN 10.....	1 ± 1	....	±20	15	25	..	..
RN 11.....	3½ ± 1½	....	±20	15	25	..	..
RN 12.....	7 ± 2	....	±20	20	25	..	..
RN 13.....	11 ± 2	....	±20	25	25	..	..
RN 14.....	15 ± 2	....	±20	25	25	..	..
RN 15.....	20½ ± 3½	....	±20	25	25	..	..
LATEX FOAM RUBBERS (CORED)							
RN 21.....	....	0.19 ± 0.07	±20	25	75	10	..
RN 22.....	....	0.35 ± 0.09	±20	25	75	10	..
RN 23.....	....	0.57 ± 0.13	±20	25	75	10	..
RN 24.....	....	0.85 ± 0.15	±20	25	75	10	..
RN 25.....	....	1.17 ± 0.17	±20	25	75	10	..
RN 26.....	....	1.53 ± 0.19	±20	25	75	10	..
RN 27.....	....	1.93 ± 0.21	±20	25	75	10	..
RN 28.....	....	2.37 ± 0.23	±20	25	75	10	..
LATEX FOAM RUBBERS (UNCORED)							
RN 31.....	....	0.15 ± 0.05	±20	25	75	..	..
RN 32.....	....	0.35 ± 0.15	±20	25	75	..	..
RN 33.....	....	0.65 ± 0.15	±20	25	75	..	..
RN 34.....	....	1.25 ± 0.45	±20	25	75	..	..
EXPANDED RUBBERS							
RN 41.....	3½ ± 1½	....	....	..	..	..	10
RN 42.....	7 ± 2	....	....	..	..	..	10
RN 43.....	11 ± 2	....	....	..	..	..	10

<sup>a</sup> These values calculated from those obtained using a 50-sq.-in. indenter foot.

shall have a natural skin on both the top and bottom surfaces. Fabric surface impressions are ordinarily not objectionable. The coarseness of the impressions shall be agreed upon by the parties concerned.

(b) *Latex Foam Rubbers*.—The struc-

ture of latex foam rubbers consists of a network of open or interconnecting cells. Latex foam rubbers may be either cored or not, as specified. Size, shape, and distribution of coring shall be at the producers option but subject to the approval of the purchaser.

TABLE II.—PHYSICAL REQUIREMENTS OF CELLULAR RUBBERS, TYPE R, CLASS RS, NON-OIL RESISTANT.

(See Section 10 for Methods of Testing)

Basic Requirements			Requirements Added by Suffix Letters				
Grade Number	Compression Deflection, 25 per cent Deflection, (Limits), psi.	Indentation, 25 per cent Deflection, (Limits), psi*	Suffix A	Suffix B	Suffix F	Suffix H	Suffix L
			Oven Aged 7 days at 158 F., Change from Original Load-Deflection or Indentation Values (Limits), per cent	Compression-Set, 22 hr. at 158 F., 50 per cent Deflection, max., per cent	Change From Original Deflection, max., per cent	Flexing Test, Compression-Set, max., per cent	Water Absorption, max., per cent
SPONGE RUBBERS							
RS 10.....	1 ± 1	....	±20	15	25	..	..
RS 11.....	3½ ± 1½	....	±20	15	25	..	..
RS 12.....	7 ± 2	....	±20	20	25	..	..
RS 13.....	11 ± 2	....	±20	25	25	..	..
RS 14.....	15 ± 2	....	±20	25	25	..	..
RS 15.....	20½ ± 3½	....	±20	25	25	..	..
LATEX FOAM RUBBERS (CORED)							
RS 21.....	....	0.19 ± 0.07	±20	25	75	10	..
RS 22.....	....	0.35 ± 0.09	±20	25	75	10	..
RS 23.....	....	0.57 ± 0.13	±20	25	75	10	..
RS 24.....	....	0.85 ± 0.15	±20	25	75	10	..
RS 25.....	....	1.17 ± 0.17	±20	25	75	10	..
RS 26.....	....	1.53 ± 0.19	±20	25	75	10	..
RS 27.....	....	1.93 ± 0.21	±20	25	75	10	..
RS 28.....	....	2.37 ± 0.23	±20	25	75	10	..
LATEX FOAM RUBBERS (UNCORED)							
RS 31.....	....	0.15 ± 0.05	±20	25	75	..	..
RS 32.....	....	0.35 ± 0.15	±20	25	75	..	..
RS 33.....	....	0.65 ± 0.15	±20	25	75	..	..
RS 34.....	....	1.25 ± 0.45	±20	25	75	..	..
EXPANDED RUBBERS							
RS 41.....	3½ ± 1½	....	....	..	..	..	10
RS 42.....	7 ± 2	....	....	..	..	..	10
RS 43.....	11 ± 2	....	....	..	..	..	10

\* These values calculated from those obtained using a 50-sq. in. indenter foot.

TABLE III.—PHYSICAL REQUIREMENTS OF CELLULAR RUBBERS, TYPE S, CLASS SB, OIL RESISTANT LOW SWELL.

(See Section 10 for Methods of Testing)

Basic Requirements				Requirements Added by Suffix Letters				
Grade Number	Compression Deflection, 25 per cent Deflection (Limits), psi.	Indentation, 25 per cent Deflection (Limits), psi.	Oil Aged 22 hr. at 158 F., Change in Volume, 160 F. AP Oil (Limits)	Suffix A	Suffix B	Suffix F	Suffix H	Suffix L
				Oven Aged 7 days at 158 F., Change from Original Load-Deflection or Indentation Values (Limits), per cent	Compression-Set, 22 hr. at 158 F., 50 per cent Deflection, max., per cent	Change From Original Deflection Values, max., per cent	Flexing Test, Compression-Set, max., per cent	Water Absorption, max., per cent
SPONGE RUBBERS								
SB 10.....	1 ± 1	....	-25 to +10	±20	15	50	..	..
SB 11.....	3½ ± 1½	....	-25 to +10	±20	15	50	..	..
SB 12.....	7 ± 2	....	-25 to +10	±20	20	50	..	..
SB 13.....	11 ± 2	....	-25 to +10	±20	25	50	..	..
SB 14.....	15 ± 2	....	-25 to +10	±20	25	50	..	..
SB 15.....	20½ ± 3½	....	-25 to +10	±20	25	50	..	..

(c) *Expanded Rubbers*.—Closed-cell rubbers are made by subjecting the compound to a gas such as nitrogen under high pressure. Under these conditions a certain amount of gas dissolves in the compound. When the pressure is low-

from slab or solid rubbers and are manufactured in sheet, strip, molded, or special shapes.

#### Types of Cellular Rubbers

4. These specifications cover two types

TABLE IV.—PHYSICAL REQUIREMENTS OF CELLULAR RUBBERS, TYPE S, CLASS SC, OIL RESISTANT, MEDIUM SWELL.

(See Section 10 for Methods of Testing)

Basic Requirements				Requirements Added by Suffix Letters					
Grade Number	Compression Deflection, 25 per cent Deflection (Limits), psi.	Indentation, 25 per cent Deflection (Limits), psi. <sup>a</sup>	Oil Aged 22 hr. at 158 F., Change in Volume, (Limits)	Suffix A Oven Aged 7 days at 158 F., Change from Original Load-Deflection or Indentation Values (Limits), per cent	Suffix B Compression-Set, 22 hr. at 158 F., 50 per cent Deflection, max., per cent	Suffix B-1 Compression-Set, 22 hr. at 158 F., 50 per cent Deflection, max., per cent	Suffix F Change from Original Deflection Values, max., per cent	Suffix H Flexing Test, Compression-Set, max., per cent	Suffix L Water Absorption, max., per cent
SPONGE RUBBERS									
			No. 3 Oil <sup>b</sup>						
SC 10.	1 ± 1	....	+10 to +60	±20	40	20	50	..	..
SC 11.	3½ ± 1½	....	+10 to +60	±20	40	20	50	..	..
SC 12.	7 ± 2	....	+10 to +60	±20	40	20	50	..	..
SC 13.	11 ± 2	....	+10 to +60	±20	40	20	50	..	..
SC 14.	15 ± 2	....	+10 to +60	±20	40	20	50	..	..
SC 15.	20½ ± 3½	....	+10 to +60	±20	40	20	50	..	..
LATEX FOAM RUBBERS (CORED)									
			No. 2 Oil <sup>b</sup>						
SC 21.	....	0.19 ± 0.07	+10 to +50	±20	25	..	75	10	..
SC 22.	....	0.35 ± 0.09	+10 to +50	±20	25	..	75	10	..
SC 23.	....	0.57 ± 0.13	+10 to +50	±20	25	..	75	10	..
SC 24.	....	0.85 ± 0.15	+10 to +50	±20	25	..	75	10	..
SC 25.	....	1.17 ± 0.17	+10 to +50	±20	25	..	75	10	..
SC 26.	....	1.53 ± 0.19	+10 to +50	±20	25	..	75	10	..
SC 27.	....	1.93 ± 0.21	+10 to +50	±20	25	..	75	10	..
SC 28.	....	2.37 ± 0.23	+10 to +50	±20	25	..	75	10	..
LATEX FOAM RUBBERS (UNCORED)									
			No. 2 Oil <sup>b</sup>						
SC 31.	....	0.15 ± 0.05	+10 to +50	±20	25	..	75	..	..
SC 32.	....	0.35 ± 0.15	+10 to +50	±20	25	..	75	..	..
SC 33.	....	0.65 ± 0.15	+10 to +50	±20	25	..	75	..	..
SC 34.	....	1.25 ± 0.45	+10 to +50	±20	25	..	75	..	..

<sup>a</sup> These values calculated from those obtained using a 50-sq. in. indenter foot.

<sup>b</sup> See Table I, Tentative Methods of Testing Cellular Rubber Products (A.S.T.M. Designation: D 552).<sup>\*</sup>

ered the mass expands and the product consists of myriads of individual non-connecting gas-tight cells. They may also be made by incorporating gas-forming ingredients in the rubber compound. Expanded rubbers are made

of cellular rubbers designated by the prefix letters R and S, as follows:

*Type R*.—Cellular rubbers made from natural rubber, reclaimed rubber, synthetic rubber, or rubber-like materials, alone or in combination, having no specific requirements for resistance to the

<sup>\*</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

action of petroleum base oils or other organic fluids.

*Type S.*—Cellular rubbers made from synthetic rubber or rubber-like materials alone, or in combination, having specific requirements for resistance to the action of petroleum-base oils or other organic fluids.

### Classes and Grades of Cellular Rubbers

5. (a) *Classes.*—Type R cellular rubbers are divided into two classes designated by the letter N and S added to the R prefix, and Type S into two classes designated by the letters B and C added to the S prefix, as follows:

*Type R: Class RN.*—Cellular rubbers containing natural rubber hydrocarbon either in the form of crude or reclaimed rubber to the extent of 50 per cent or more by volume of the total elastomer content. *No specified requirement for oil resistance.* *Class RS.*—Cellular rubbers containing synthetic rubber or rubber-like materials to the extent of 50 per cent or more by volume of the total elastomer content. *No specified requirements for oil resistance.*

*Type S: Class SB.*—Cellular rubbers made from synthetic rubber or rubber-like materials having *oil resistance with low swell.* *Class SC.*—Cellular rubbers made from synthetic rubber or rubber-like materials having *oil resistance with medium swell.*

(b) *Grades.*—Each class of cellular rubbers has a number of different grades. The grades shall be designated by numbers following prefix letters.

*Grade Numbers* shall consist of two digits, the first of which identifies the kind of cellular rubbers as follows:

1. Sponge rubbers,
2. Latex foam rubbers, cored,
3. Latex foam rubbers, uncured, and
4. Expanded rubbers.

The second digit is used to indicate the

degree of firmness of the cellular rubbers, the softer grades being identified with the lower numbers and the firmer grades being identified with the higher numbers (see Tables I to IV).

*Suffix Letters* may be added singly or in combination after any grade numbers to indicate additional requirements beyond those specified in Tables I to IV as basic requirements for that particular grade. The significance of the approved suffix letters is as follows:

### SUFFIX LETTER

- |       |   |  |
|-------|---|--|
| A.... | { | Oven aging test is required with the values as specified in Tables I, II, III, and IV.                   |
| B.... | { | Compression set test required with values as specified in Tables I, II, III, and IV.                     |
| C.... | { | Weather resistance required. Test to be arranged between the manufacturer and the purchaser.             |
| F.... | { | Grades passing the low-temperature test at -40 F. with values as specified in Tables I, II, III, and IV. |
| H.... | { | Flexing test required with values as specified in Tables I, II, and IV.                                  |
| L.... | { | Water absorption test required with values as specified in Tables I and II.                              |
| Z.... |   | Performance or special test required.  |

NOTE: *Example.*—Grade RN 11 AF denotes extra soft sponge rubber containing rubber either in the form of natural crude, or reclaimed rubber to the extent of 50 per cent or more by volume of the total elastomer content with a load deflection value of  $3\frac{1}{2} \pm 1\frac{1}{2}$  psi., and requiring in addition to the basic tests an oven aging test and a low-temperature test at -40 F.

### Material and Workmanship

6. Cellular rubbers furnished under these specifications shall be manufactured from natural rubber, synthetic rubber, or rubber-like materials together with added compounding ingredients of such nature and quality that the finished

product complies with the specification requirements. In permitting choice in use of those materials by the producer, it is not intended to imply that the different rubber materials are equivalent in respect to all physical properties. Any special characteristics other than those prescribed in these specifications which may be desired for specific applications shall be specified in the product specifications as they may influence the choice of the type of rubber material or other ingredients used. All materials and workmanship shall be in accordance with good commercial practice, and the resulting cellular rubbers shall be free from defects affecting serviceability.

#### Color

7. Unless otherwise specified, the color of cellular rubbers shall be optional with the manufacturer.

#### Physical Properties

8. The various grades of cellular rubber shall conform to the requirements as to physical properties prescribed in Tables I, II, III, and IV, together with any additional requirements indicated by suffix letters in the grade designations as described in Section 5.

#### Sampling

9. The sampling procedure shall conform to the procedure described in the Tentative Methods of Testing Cellular Rubber Products (A.S.T.M. Designation: D 552).<sup>3</sup> When the form in which the material is furnished is unsuitable for the preparation of the test specimens required, the manufacturer shall furnish standard test specimens conforming to the requirements prescribed in A.S.T.M. Methods D 552.

#### Methods of Testing

10. Unless specifically stated otherwise, all tests shall be made in accordance with the Tentative Methods of Testing

Cellular Rubber Products (A.S.T.M. Designation: D 552).<sup>3</sup>

#### Tolerances on Dimensions

11. Tolerances on dimensions of cellular rubber products are given in the Appendix (Tables V and VI). These tolerances are published as information for guidance only and shall not be considered as a part of these specifications for cellular rubber products.

#### Packaging and Marking

12. The material shall be properly and adequately packaged. Each package or container shall be legibly marked with the name of the material, name or trademark of the manufacturer, and any required purchaser's designations.

#### Inspection and Rejection

13. (a) All tests and inspection shall be made at the place of manufacture prior to shipment, unless otherwise specified. The manufacturer shall afford the inspector all reasonable facilities, without charge, for tests and inspection.

(b) The purchaser may make the tests and inspection to govern acceptance or rejection of the material at his own laboratory or elsewhere. Such tests and inspection shall be made at the expense of the purchaser, and not later than 15 days after receipt of the material.

(c) All samples for testing, provided as specified in Section 9, shall be visually inspected to determine compliance with the material, workmanship, and color requirements.

(d) Any material which fails in one or more of the test requirements may be retested at the expense of the manufacturer. For this purpose, two additional tests shall be made for the requirement in which failure occurred. Failure of either of the retests shall be cause for final rejection.

(e) Rejected material shall be disposed of as directed by the manufacturer and at his expense.



# APPENDIX

## TOLERANCES ON DIMENSIONS OF CELLULAR RUBBER PRODUCTS

The tolerances on dimensions of cellular rubber products shown in Tables V and VI are for guidance only and shall not be considered as part of these specifications.

TABLE V.—TOLERANCES ON DIMENSIONS OF CELLULAR RUBBER PRODUCTS FOR GENERAL APPLICATIONS.

	Thickness, in.			Length and Width, in.		
	Dimension	Tolerance		Dimension	Tolerance	
		Plus	Minus		Plus	Minus
SPONGE RUBBERS						
Sheet and strip	$\frac{1}{8}$ and under.....	$\frac{1}{16}$	$\frac{1}{16}$	6 and under.....	$\frac{1}{16}$	$\frac{1}{16}$
	Over $\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	$\frac{1}{32}$	$\frac{1}{32}$	Over 6 to 18, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
	Over $\frac{1}{2}$ .....	$\frac{3}{64}$	$\frac{3}{64}$	Over 18.....	$\frac{1}{4}$	$\frac{1}{4}$
Molded or special shapes..	$\frac{1}{8}$ and under.....	$\frac{1}{32}$	$\frac{1}{32}$	$\frac{1}{4}$ and under.....	$\frac{1}{32}$	$\frac{1}{32}$
	Over $\frac{1}{4}$ to 3, incl.....	$\frac{1}{16}$	$\frac{1}{16}$	Over $\frac{1}{4}$ to 3, incl.....	$\frac{1}{16}$	$\frac{1}{16}$
				Over 3 to 6, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
			Over 6.....	$\frac{1}{4}$	$\frac{1}{4}$	
LATEX FOAM RUBBERS						
Cored.....	0 to 3, incl.....	$\frac{1}{8}$	$\frac{1}{16}$	0 to 6, incl.....	$\frac{3}{16}$	$\frac{1}{16}$
	3 to 5, incl.....	$\frac{3}{16}$	$\frac{1}{8}$	6 to 12, incl.....	$\frac{1}{2}$	$\frac{1}{8}$
	5 and over.....	$\frac{1}{4}$	$\frac{3}{16}$	12 to 24, incl.....	$\frac{1}{2}$	$\frac{1}{4}$
				24 to 36, incl.....	$\frac{5}{8}$	$\frac{3}{8}$
				36 to 48, incl.....	$\frac{3}{4}$	$\frac{1}{2}$
				48 to 60, incl.....	$\frac{7}{8}$	$\frac{3}{4}$
				60 to 72, incl.....	1	$\frac{3}{4}$
			72 and over.....	$1\frac{1}{8}$	$\frac{3}{8}$	
Uncored.....	$\frac{1}{4}$ to $\frac{3}{8}$ , incl.....	$\frac{1}{8}$	$\frac{1}{16}$	0 to 6, incl.....	$\frac{5}{16}$	$\frac{1}{16}$
	$\frac{3}{8}$ to $\frac{1}{2}$ , incl.....	$\frac{1}{8}$	$\frac{1}{8}$	6 to 12, incl.....	$\frac{1}{2}$	$\frac{1}{8}$
	$\frac{1}{2}$ to 1, incl.....	$\frac{3}{8}$	$\frac{1}{8}$	12 to 24, incl.....	$1\frac{1}{16}$	$\frac{1}{4}$
	$\frac{3}{4}$ to 1, incl.....	$\frac{5}{8}$	$\frac{1}{8}$	24 to 36, incl.....	$1\frac{1}{8}$	$\frac{3}{8}$
	$1\frac{1}{4}$ and over.....	$\frac{1}{4}$	$\frac{3}{16}$	36 to 48, incl.....	$1\frac{1}{8}$	$\frac{1}{2}$
				48 to 60, incl.....	$1\frac{3}{4}$	$\frac{5}{8}$
				60 to 72, incl.....	$1\frac{5}{8}$	$\frac{3}{4}$
			72 and over.....	$1\frac{1}{2}$	$\frac{7}{8}$	
EXPANDED RUBBERS						
Sheet and strip	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	$\frac{1}{16}$	$\frac{1}{16}$	6 and under.....	$\frac{1}{4}$	$\frac{1}{4}$
	Over $\frac{1}{2}$ .....	$\frac{3}{32}$	$\frac{3}{32}$	6 to 12, incl.....	$\frac{3}{8}$	$\frac{3}{8}$
				Over 12.....	3 per cent	3 per cent
Molded or special shapes..	$\frac{1}{8}$ to $\frac{1}{2}$ , incl.....	$\frac{1}{16}$	$\frac{1}{16}$	6 and under.....	$\frac{1}{4}$	$\frac{1}{4}$
	$\frac{1}{2}$ to $1\frac{1}{2}$ , incl.....	$\frac{3}{32}$	$\frac{3}{32}$	6 to 12, incl.....	$\frac{3}{8}$	$\frac{3}{8}$
	$1\frac{1}{2}$ to 3, incl.....	$\frac{1}{8}$	$\frac{1}{8}$	Over 12.....	3 per cent	3 per cent

TABLE VI.—TOLERANCES FOR SPECIAL APPLICATIONS OF LATEX FOAM RUBBERS, SUCH AS AUTOMOTIVE TOPPER PADS, SPRING COVERINGS, ETC.

	Thickness, in.			Length and Width, in.		
	Dimension	Tolerance		Dimension	Tolerance	
		Plus	Minus		Plus	Minus
Cored.....	0 to 3, incl.....	$\frac{3}{16}$	$\frac{1}{16}$	0 to 6, incl.....	$\frac{5}{16}$	$\frac{1}{16}$
	3 to 5, incl.....	$\frac{1}{4}$	$\frac{3}{8}$	6 to 12, incl.....	$\frac{3}{8}$	$\frac{1}{8}$
	5 and over.....	$\frac{1}{8}$	$\frac{1}{16}$	12 to 24, incl.....	$\frac{1}{16}$	$\frac{1}{4}$
				24 to 36, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
				36 to 48, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
				48 to 60, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
Uncored.....	$\frac{1}{4}$ to $\frac{3}{4}$ , incl.....	$\frac{1}{8}$	$\frac{1}{16}$	0 to 6, incl.....	$\frac{5}{16}$	$\frac{1}{16}$
	$\frac{1}{2}$ to $\frac{3}{2}$ , incl.....	$\frac{1}{8}$	$\frac{3}{8}$	6 to 12, incl.....	$\frac{3}{8}$	$\frac{1}{8}$
	$\frac{3}{4}$ to 1, incl.....	$\frac{3}{16}$	$\frac{1}{8}$	12 to 24, incl.....	$\frac{1}{16}$	$\frac{1}{4}$
	$1\frac{1}{4}$ and over.....	$\frac{1}{4}$	$\frac{3}{8}$	24 to 36, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
				36 to 48, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
				48 to 60, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
				60 to 72, incl.....	$\frac{1}{8}$	$\frac{1}{8}$
				72 and over.....	$\frac{1}{8}$	$\frac{1}{8}$

# *Tentative Methods of*

## TESTING HARD RUBBER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 530 - 44 T

ISSUED, 1939; REVISED, 1939, 1944.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These methods are intended for testing hard rubber products and are classified for the purpose of this standard into three separate procedures:

Chemical Analysis,  
Physical Tests, and  
Electrical Tests.

(b) The term "hard rubber" as used herein shall be understood to refer to vulcanized rubber compositions having a ratio of combined sulfur to rubber hydrocarbon in excess of 15 per cent.

(c) It is intended that the procedures described in these methods shall be used only as required for measurement of specified properties. It shall not be inferred that all included tests are necessary for every product.

(d) In case of conflict between the provisions of these methods and those of detailed specifications or methods of test for a particular hard rubber product, the latter shall take precedence.

### CHEMICAL ANALYSIS

#### Chemical Analysis

2. The chemical analysis of hard rubber shall be made in accordance with the complete procedure as described in Sections 2 to 34 and Section 47 of the Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297) of the American Society for Testing Materials.<sup>3</sup>

### PHYSICAL TESTS

#### Description of Terms

3. (a) *Tensile Strength*.—Tensile strength is the tension load per unit cross-sectional area required to break a test specimen of the type and in the manner described in these methods.

(b) *Elongation*.—Elongation is the extension produced by a tension load in a section of a test specimen between gage marks placed on it, expressed as a percentage of the original distance between the marks. Ultimate elongation is the elongation at the moment of rupture.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Latest revision accepted by Committee E-10 on Standards, December 13, 1944.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(c) *Flexural Strength*.—Flexural strength is the transverse load per unit cross-sectional area required to break a test specimen of the type and in the manner described in these methods.

(d) *Impact Resistance*.—Impact resistance is the energy absorbed in breaking a test specimen of the type and in the manner described in these methods, expressed in terms of foot-pounds per unit cross-sectional area.

(e) *Hardness*.—Hardness is an arbitrary measurement of the resistance to penetration by a ball under a dead weight, under the conditions of test specified.

(f) *Cold Flow*.—Cold flow is the relative deformation caused by the application of a prescribed load on the test specimen in the manner described in these methods.

(g) *Softening Point*.—The softening point is a temperature value, indicating in a relative manner the resistance to bending under a constant load at elevated temperatures. Since hard rubber has no definite softening point, this is an arbitrary value depending upon the manner in which the test is conducted.

### Test Specimens

4. (a) If a section of sufficient size is available, the test specimens shall be cut from the product to be tested. Otherwise the test specimens may be cut from an especially prepared test piece which has been vulcanized under the same conditions as the product.

(b) If the material has been calendered in processing, each test specimen shall be cut so that its longitudinal axis is in the direction of the calender grain, whenever possible.

(c) The test specimens shall be sawed, machined, filed, sanded, or ground to the prescribed size and shape while exercising especial care to prevent excessive heating and to obtain smooth surfaces

free of nicks, scratches, grooves, or ragged edges.

### Measurement, Aging, and Conditioning of Specimens

5. (a) The thickness and width of each specimen shall be measured with a micrometer as specified in the Standard Methods of Test for Thickness of Solid Electrical Insulation (A.S.T.M. Designation: D 374) of the American Society for Testing Materials,<sup>3</sup> or with a micrometer dial gage, graduated to 0.001 in. having a presser foot  $0.25 \pm 0.01$  in. in diameter exerting a total force of  $3.0 \pm 0.1$  oz. The load shall be applied by a weight.

(b) The minimum thickness and width shall be used in calculating the cross-sectional area.

(c) Tests shall not be made until specimens have aged at least 24 hr. after vulcanization. At least 16 hr. but not more than 36 hr. shall elapse between the time of preparation of the specimen and the time of testing. The specimens shall be immersed in water for at least 1 hr. at  $70 \pm 2$  F. ( $21.1 \pm 1$  C.) before testing and shall be tested without drying.

### Temperature of Test

6. All tests, except as otherwise specified, shall be made at a room temperature between 70 and 90 F. ( $21.1$  and  $32.2$  C.).

### TENSILE STRENGTH AND ELONGATION

#### Apparatus

7. (a) *Testing Machine*.—Tensile strength and elongation shall be determined on a power-driven apparatus of either the beam-weighing or pendulum type conforming to the following requirements:

The applied tension as indicated by a

dial or scale shall be accurate within 1 per cent.

The indicator shall remain at the point of maximum load after rupture of the test specimen.

The rate of travel of the power-actuated grip shall be 0.4 in. per min. and shall be uniform at all times.

The testing machine used for a given test specimen shall be of such capacity that the maximum load required to break the specimen shall not exceed 85 per cent nor be less than 15 per cent of the rated capacity. For the tests described in these methods, a machine of 1000-lb. maximum capacity will be satisfactory.

(b) *Grips*.—The grips which hold the specimen in the testing machine may be of a wedge type which tighten automatically and exert a uniform pressure across the gripping surface proportional to the applied tension, or of the type which may be tightened by screw pressure by hand. The gripping surfaces shall be knurled to prevent slippage of the test specimen.

#### Calibration of Testing Machine

8. The testing machine shall be calibrated by dead weights applied in an ascending order while the machine is otherwise arranged in an entirely similar manner to that used when testing material. In each case when an additional calibrating weight is added, the weight lever of the machine shall be moved to zero position and allowed to swing slowly upwards to the point where it will go no further. The dial of the machine should then correctly indicate the amount of the weight applied.

#### Test Specimen

9. (a) The standard specimen shall conform in shape to Fig. 1, although a variation in width of plus 0.005 in. or minus 0.025 in. shall be permitted. The variation in width of an individual

specimen shall be not greater than 0.005 in. The thickness shall be  $0.125 \pm 0.005$  in.

(b) Two parallel gage lines 3 in. apart, for use in determining elongation, shall be scratched on the reduced section of the specimen by means of a sharp pointed instrument such as a pair of dividers. The lines shall be perpendicular to the longitudinal axis of the specimen, one on each side of the center and 1.5 in. therefrom. The marks shall be as fine as possible, consistent with legibility, and shall not be deep enough to cause a weakening of the specimen.

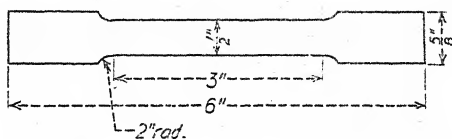


FIG. 1.—Tension Test Specimen.

#### Number of Specimens

10. Three specimens per sample shall be tested for determination of tensile strength and elongation. Results on specimens which break outside of the straight reduced section or on specimens which are obviously defective shall be discarded and retests shall be made.

#### Procedure

11. The test specimen shall be placed in the grips using care to adjust it symmetrically in order that the tension shall be distributed uniformly over the cross-section. Tension load shall be applied to the specimen by the power-actuated grip which shall travel at the specified rate of speed until rupture takes place. During the load application, the distance between the gage marks on the specimen shall be noted continuously by means of dividers so that the distance at the instant of rupture may be determined within 0.01 in. This value shall be recorded for use in calculating the

ultimate elongation. After rupture of the specimen, the breaking load in pounds shall be noted from the scale and recorded together with the original minimum width and thickness of the specimen so that the tensile strength may be calculated.

### Calculations

12. (a) Tensile strength shall be calculated by dividing the maximum load in pounds by the original area of cross-section of the test specimen in square inches and shall be expressed in pounds per square inch.

(b) Ultimate elongation shall be calculated by subtracting the original distance between gage marks in inches (3 in.) from the total distance between the marks at the time of rupture as measured in Section 11, and expressing this difference as a percentage of the original distance, as follows:

$$E = \frac{d - 3.0}{3.0} \times 100$$

where:

$E$  = ultimate elongation in per cent,  
and

$d$  = distance between gage marks on the specimen at the time of rupture.

### Report

13. The report shall include the following:

(a) The results calculated in accordance with Section 12,

(b) All observed and recorded data on which the calculations are based,

(c) The date of vulcanization, if known,

(d) The temperature of the test room,

(e) The type of test machine used, and

(f) The date of test.

### FLEXURAL STRENGTH

#### Apparatus

14. (a) *Testing Machine*.—The testing machine shall be of the type described in Section 7 and shall meet the requirements there specified.

(b) *Bearings*.—The flexing apparatus shall consist of two hard-metal supports and one pressure piece. The distance between the points of support shall be fixed at 3 in. The supports and pressure piece shall have bearing edges rounded to a radius of not less than 0.125 in. and not more than 0.5 in. and shall be wider than the width of the test specimen. The supports and bearing piece shall be mounted in the testing machine so that all edges of the pieces are parallel.

(c) *Scale*.—Either an extensometer or a dial scale graduated to 0.01 in. may be used to read the deflection.

#### Test Specimen

15. The specimen shall be 4.75 in. in length by 0.875 in. in width by 0.1875 in. in thickness, although a variation in width and thickness of plus 0.005 in. and minus 0.025 in. shall be permitted. The variation in width and thickness of an individual specimen shall not be greater than 0.005 in.

#### Number of Specimens

16. Three specimens per sample shall be tested. Results on specimens which are obviously defective shall be discarded and retests shall be made.

#### Procedure

17. The test specimen shall be centered with the wide face on the supports. The load shall be applied perpendicularly to the specimen and midway between the supports, until failure. During this period the load and deflection shall be noted continuously so that the load in

pounds and deflection in inches are known at the time of rupture.

#### Calculation

18. The maximum fiber stress in pounds per square inch shall be calculated from the following formula:

$$S = \frac{3Pl}{2bd^3}$$

where:

- $S$  = maximum fiber stress,
- $P$  = load at rupture in pounds,
- $l$  = distance between points of support in inches,
- $b$  = width of specimen as tested in inches, and
- $d$  = depth of specimen as tested in inches.

#### Report

19. The report shall include the following:

- (a) The thickness and width of each specimen,
- (b) The load on each specimen in pounds at the time of failure,
- (c) The maximum fiber stress,
- (d) The maximum deflection at the center in hundredths of an inch,
- (e) The date of vulcanization, if known,
- (f) The temperature of the test room,
- (g) The type of test machine used, and
- (h) The date of test.

#### IMPACT RESISTANCE

##### Procedure

20. The impact resistance shall be determined in accordance with the Tentative Methods of Test for Impact Resistance of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 256) of the American Society for Testing Materials<sup>3</sup> with the following exceptions:

- (a) The test specimens may be cut

from the product if the product is of sufficient size.

(b) Conditioning of the test specimens as prescribed in Sections 4 and 9 of Methods D 256 is not required.

(c) The temperature of the test room shall also be reported.

#### HARDNESS

##### Nature of Test

21. In the Rockwell test, although the value given on the dial depends on the actual depth of penetration, the hardness figure is a measurement of the vertical motion of the plunger rod which carries the penetrator as that plunger rod moves up and down in the head of the machine. This makes the depth measurement quite independent of the behavior of the surface in the vicinity of the penetration. Another important feature is that the dial gage is not set to zero when the penetrator first meets the surface of the material to be tested. Instead there is applied a load of 10 kg. called the minor load, which is applied before the dial gage is set for zero. This means that the Rockwell hardness number, instead of being based upon the total depth of penetration, is based upon the increment of depth, due to increment of load from minor load to major load.

##### Apparatus

22. The Rockwell hardness tester shall conform to the requirements for the apparatus prescribed in the Standard Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials (A.S.T.M. Designation: E 18) of the American Society for Testing Materials,<sup>4</sup> with the exception that a 60-kg. major load shall be applied to the specimen using a  $\frac{1}{4}$ -in. ball penetrator.

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Parts I-A and I-B.

### Test Specimen

23. The specimen shall have a minimum thickness of 0.125 in.

### Procedure

24. (a) The test specimen shall be placed on the anvil selected to provide the best balance of the specimen. The specimen shall be supported in such a manner as to prevent errors due to overhang, when it does not balance itself on the anvil. For small flat specimens or flat specimens that can be readily balanced the 0.125-in. spot anvil shall be used; for larger flat specimens the 3-in. (or larger) flat anvil shall be used. For specimens in rod or tube form (Note 1) a V-shaped anvil shall be used and in the case of tubing a supporting mandrel shall be inserted in the hole so as to prevent error due to springing under the major load.

NOTE 1.—The hardness values of rod and tubing determined in accordance with Section 24 (a) are only relative for control purposes and can not be correlated in terms of a standard hardness value obtained on flat specimens.

(b) The 10-kg. minor load shall be applied by raising the pedestal until the small pointer is at the red dot (on the face of the dial) and the indicator needle is in a vertical position. The scale shall be turned so that the needle is at B30 on the red scale. The indicator needle may be stopped within five divisions on either side of the vertical position and the scale shall be adjusted to bring the zero point in position with the needle.

(c) The major load shall be applied (Note 2) immediately and maintained for 15 sec., at which time the travel of the needle in scale divisions shall be read. The major load shall then be removed and 15 sec. later, the recovery, or travel of the needle in the reverse direction, shall be read. These readings shall be recorded as the Rockwell penetration and Rockwell recovery values, respectively (Notes 3 and 4).

NOTE 2.—Care shall be taken that the test is not made so near the edge of the specimen that it will spread or break out when the major load is applied.

NOTE 3.—This method is not applicable to semi-hard materials of which the Rockwell penetration value is over 200.

NOTE 4.—The HR Rockwell hardness number, formerly used, is equal to the Rockwell recovery value plus 130 minus the Rockwell penetration value.

### Report

25. The report shall include the following:

- (1) The Rockwell penetration value after 15 sec. under major load,
- (2) The Rockwell recovery value taken 15 sec. after removal of major load,
- (3) The thickness of the specimen,
- (4) The room temperature,
- (5) The date of vulcanization, if known, and
- (6) The date of test.

### COLD FLOW

#### Nature of Test

26. Cold flow as measured by this method is a determination of the change in thickness, expressed as a percentage of the original thickness, caused by the application of a known load at a specified temperature for a definite time. The apparatus used consists essentially of a lever arrangement capable of producing a relatively heavy loading on a standard size test specimen.

#### Apparatus

27. (a) The testing machine shall be constructed substantially as shown in Fig. 2. The ratio of the lever shall be 10 to 1. The machine shall be capable of producing a load of 1000 lb. on the test specimen.

(b) The testing machine shall be mounted in a chamber in which the temperature may be maintained at  $120 \pm 1$  F. ( $48.9 \pm 0.5$  C.).



**Test Specimen**

28. The test specimen shall be in the form of a 0.5-in. cube or a pile-up of 0.5-in. squares if the material is less than 0.3 in. in thickness. The permissible variations in dimensions, excepting thickness, shall not exceed plus or minus 0.005 in.

**Number of Specimens**

29. Two test specimens shall be tested.

**Procedure**

30. The test specimen shall be placed on the anvil of the machine without

**SOFTENING POINT****Apparatus<sup>4a</sup>**

32. The testing machine shall be the same as that used for the distortion under heat test as described in the Tentative Method of Test for Heat Distortion Temperature of Plastics (A.S.T.M. Designation: D 648) of the American Society for Testing Materials.<sup>3</sup>

**Test Specimen**

33. The test specimen shall be 4.75 in. in length, 0.5 in. in width and 0.1875 in. in thickness. A variation of plus or

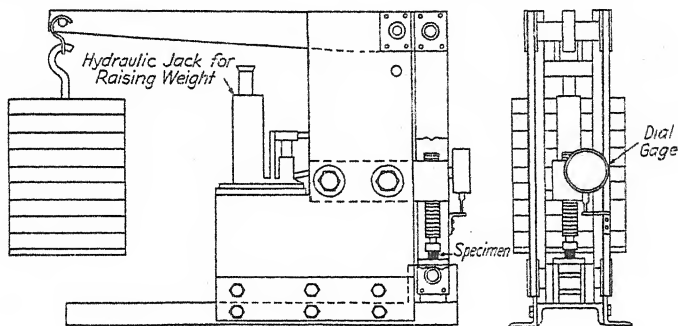


FIG. 2.—Cold Flow Testing Machine.

previous heating and the thickness read from the dial gage at the time the load is applied. The thickness shall be read again after subjecting the test specimen to a load of 1000 lb. for 24 hr., at 120 F. (48.9 C.). The difference in thickness, expressed as a percentage of the original thickness, shall be reported as cold flow.

**Report**

31. The report shall include the following:

- (a) The cold flow expressed in per cent,
- (b) The original thickness,
- (c) The date of vulcanization, if known, and
- (d) The date of test.

minus 0.005 in. shall be permitted in the width and thickness.

**Number of Specimens**

34. Two test specimens shall be tested and the average values shall be used in determining the softening point.

**Procedure**

35. The apparatus shall be at room temperature when the test is started. The test specimen shall be placed on the supports and the load applied. After the gage has come to rest it shall be reset to zero. The heat shall then be applied

<sup>4a</sup> Editorially revised, March, 1946.

to increase the temperature gradually at a rate of 3.5 F. (2 C.) per min. The temperature shall be recorded for each deflection of 0.015 in. The tests shall be continued until the deflection increases so rapidly that it is difficult to read the corresponding temperature. A curve shall then be plotted of deflection *versus* temperature and a tangent shall be drawn to the linear portion of the curve. The softening point shall be the temperature indicated by the intersection of the tangent and the temperature axis.

NOTE.—This test is designed particularly for hard, heat-resistant rubber compounds and is not suitable for semihard compounds.

### Report

36. The report shall include the following:

- (a) The softening point,
- (b) The date of vulcanization, if known, and
- (c) The date of test.

## ELECTRICAL TESTS

### Dielectric Strength

37. The dielectric strength of hard rubber shall be determined in accordance with the Standard Methods of Test for Dielectric Strength of Electrical Insulating Materials at Commercial Power Frequencies (A.S.T.M. Designation: D 149) of the American Society for Testing Materials.<sup>3</sup>

### Power Factor

38. The power factor of hard rubber shall be determined in accordance with the Tentative Methods of Test for Power Factor and Dielectric Constant of Electrical Insulating Materials (A.S.T.M. Designation: D 150) of the American Society for Testing Materials.<sup>3</sup>

### Surface and Volume Resistivity

39. The surface and volume resistivity of hard rubber shall be determined in accordance with Standard Methods of Test for Insulation Resistance of Electrical Insulating Materials (A.S.T.M. Designation: D 257) of the American Society for Testing Materials,<sup>3</sup> except that the use of tin foil, sputtered tin, or colloidal graphite electrodes as well as the specified mercury electrodes shall be permitted. Tin foil electrodes which may be used for volume resistivity measurements are described in Section 13 (a) of Methods D 150. The guard ring may be omitted provided the distance over the surface between electrodes is sufficiently great to reduce surface leakage to a negligible value. For surface resistivity measurements the space between the center electrode and the ring surrounding it shall be increased to a reasonable value such as 0.25 in. Sputtered tin or colloidal graphite electrodes for volume or surface resistivity measurements shall have the same dimensions and shape as the corresponding mercury or tin foil electrodes.

# *Tentative Methods of*

## TESTING ASPHALT COMPOSITION BATTERY CONTAINERS<sup>1</sup>



A.S.T.M. Designation: D 639 - 46 T

ISSUED, 1941; REVISED, 1943, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) These methods are intended for use in testing battery containers made of asphalt compositions, as distinguished from those made of hard rubber.

(b) Although the procedures described in these methods have been prepared expressly for the testing of asphalt composition containers, it is not to be inferred that no other tests are applicable, since tests such as dielectric strength, specific gravity, ash, etc., apply to such compositions as well as to hard rubber.

### CHEMICAL ANALYSIS

NOTE.—The development of methods for the chemical analysis of these compositions has been restricted to the determination of soluble manganese and iron, since excessive amounts of these elements, each of which is usually found in small proportions, are injurious to the performance of a battery.

### Preparation of Sample

2. The sample shall be prepared by taking pieces from various parts of the container and separating from them any foreign matter. Surface coatings, in particular, shall be scraped off before there is further division of the pieces. These pieces, preferably, shall then be mounted in a shaper machine and cuts made by feeding the tool at 0.01 in. per stroke to a depth of approximately  $\frac{1}{8}$  in. In lieu of a shaper, borings may be taken with a  $\frac{1}{8}$ -in. drill at a rate of feed that will give a sample approximately 0.01 in. in thickness and fragments that approximate in size those obtained with a shaper. Oil shall not be used on the cutting tools. If water is used as a lubricant, the material shall be dried before it is weighed. Thorough blending of the sample shall be carried out before proceeding with the analysis.

### Preparation of Acid Extract of Sample

3. Place 20 g. of the sample in a 500-ml. Erlenmeyer flask provided with a reflux condenser, and boil with 300 ml.

<sup>1</sup>Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup>Latest revision accepted by the Society at annual meeting, June, 1946.

of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.30) for 2 hr. (Note) Filter off the insoluble matter on a 24-cm. Whatman No. 2 filter paper or equivalent by means of suction, and wash with hot distilled water until all soluble sulfates are removed. Cool the filtrate and washings to room temperature, transfer to a 500-ml. volumetric flask, and dilute to volume.

NOTE.—In order to effectively remove all soluble iron and manganese, it is necessary to wet the entire sample with acid before refluxing.

#### MANGANESE BY THE PERSULFATE METHOD

##### Reagent

4. *Standard Sodium Arsenite Solution.*—Dissolve, by heating, 10 g. of c.p.  $\text{As}_2\text{O}_3$  in 100 ml. of water containing 30 g. of c.p.  $\text{Na}_2\text{CO}_3$ . Cool and dilute to 1 liter. Dilute 125 ml. of this solution to 2 l. Standardize the solution against 0.1 *N*  $\text{KMnO}_4$ .

##### Procedure

5. (a) Pipette a 250-ml. aliquot of the acid extract (Section 3) into a 400-ml. Griffin beaker. Add 10 ml. of  $\text{HNO}_3$  (sp. gr. 1.42) and heat to dense white fumes. Cool, add 2 to 3 ml. of  $\text{HNO}_3$  (sp. gr. 1.42), and again heat to dense white fumes. Repeat this treatment until further addition of  $\text{HNO}_3$  and heating do not produce further change in the appearance of the solution.

(b) Cool, add 20 to 25 ml. of  $\text{AgNO}_3$  (3 g. per l.), and dilute to 100 ml. Heat to boiling, slowly add 2 g. of solid crystalline  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , and stir after each addition. Cool to 25 C., and add 5 ml. of  $\text{NaCl}$  (2 g. per l.). Do not filter the  $\text{AgCl}$  precipitate from the solution, so as to avoid any possible reduction from the fully developed permanganate color. Titrate the solution (Note) with standard sodium arsenite solution (Section 4) until the pink color has completely disappeared. For samples containing less

than 0.00003 g. of manganese, titrate the solution with 0.005 *N*  $\text{Na}_2\text{HAsO}_3$  (Note).

NOTE.—In view of the small volumes of standard solution required in this determination, it is desirable to use a microburette.

(c) *Calculation.*—Calculate the percentage of manganese as follows:

$$\text{Manganese, per cent} = \frac{AB}{W} \times 100$$

where:

*A* = milliliters of  $\text{Na}_2\text{HAsO}_3$  solution required to titrate the sample,  
*B* = manganese equivalent of  $\text{Na}_2\text{HAsO}_3$  solution, and  
*W* = weight in grams of sample in aliquot.

#### MANGANESE BY THE PERIODATE (ALTERNATIVE METHOD)

##### Apparatus

6. *Colorimeter.*—A photocolormeter, Duboscq colorimeter, or Nessler tubes may be used for the color comparison.

##### Reagents

7. (a) *Phosphoric Acid (85 per cent).*  
 (b) *Sulfuric Acid—Potassium Periodate Solution.*—Prepare a freshly-boiled solution containing 8 per cent of  $\text{H}_2\text{SO}_4$  and 1 per cent of  $\text{KIO}_4$ .

(c) *Colorimetric Standards for Manganese.*—Prepare color standards of known permanganate content by treating definite amounts of a pure manganese salt in the same manner as described for the aliquot portion of the acid extract in Section 8. The color standards shall be freshly prepared for each test, except when a calibration curve has been established on a reliable photocolormeter.

##### Procedure

8. (a) Treat a 100-ml. aliquot of the acid extract (Section 3) as described in Section 5 (a). Cool the solution, dilute to 40 ml., and add 2 to 3 ml. of  $\text{H}_3\text{PO}_4$  (85 per cent), 5 ml. of  $\text{HNO}_3$  (sp. gr.

1.42), and 0.5 g. of  $\text{KIO}_4$ . Boil the solution gently until the permanganate color is fully developed (about 5 min.). If the color shows any tendency to fade, add 0.5 g. of  $\text{KIO}_4$ , and boil again.

(b) Cool the solution and transfer to a 50-ml. colorimeter tube. Dilute to 50 ml. with the freshly-boiled  $\text{H}_2\text{SO}_4$  -  $\text{KIO}_4$  solution, and compare with the series of color standards of known permanganate content. Because of the great sensitivity of permanganate to reduction, all mixing should be done without contact with hands, rubber stoppers, or other reducing agents.

(c) *Calculation.*—Calculate the percentage of manganese as follows:

$$\text{Manganese, per cent} = \frac{S}{W} \times 100$$

where:

$S$  = manganese content in 50 ml. of matching color standard, and  
 $W$  = weight in grams of sample in aliquot.

## IRON

### Apparatus

9. *Colorimeter.*—A Nessler tube or other suitable colorimeter may be used for the color comparison.

### Reagents

10. *Standard Iron Solution* (1 ml. = 0.0001 g. Fe).—Dissolve while heating exactly 0.1 g. of standardization grade iron wire in 300 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.30). Oxidize the iron by adding 5 ml. of bromine water, and boil off the excess bromine. Cool, and dilute in a volumetric flask to exactly 1 liter.

### Procedure

11. (a) Pipette a 250-ml. aliquot of the acid extract (Section 3) into a 400-ml. Griffin beaker (Note). Add 2 ml. of  $\text{HNO}_3$  (sp. gr. 1.42). Heat to boiling and allow to cool. Precipitate the iron with  $\text{NH}_4\text{OH}$  (sp. gr. 0.90), adding

5 ml. in excess. Warm to coagulate the  $\text{Fe}(\text{OH})_3$ . Filter and wash with hot  $\text{NH}_4\text{Cl}$  (20 g. per l.), discarding the filtrate. Redissolve the precipitated iron from the paper with 50 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.20), collecting the filtrate in a 200-ml. volumetric flask. Dilute to 200 ml. with distilled water.

(b) Pipette a 20-ml. aliquot into a 100-ml. colorimetric tube. Add 5 ml. of  $\text{NH}_4\text{CNS}$  (40 g. per l.). Dilute to 100 ml. with water, and mix.

(c) *Blank.*—Make a blank determination following the same procedure, using the same amount of diluted acid as used in the aliquot sample. Titrate the blank with standard iron solution (1 ml. = 0.0001 g. Fe), adding it drop by drop and mixing thoroughly after each addition until the depth of color of the blank and sample tubes match.

NOTE.—If the iron content of the aliquot is too high (more than 0.010 g.) the size of the sample for determination shall be reduced accordingly.

(d) *Calculation.*—Calculate the percentage of iron as follows:

$$\text{Iron, per cent} = \frac{D}{W} \times 0.01$$

where:

$D$  = milliliters of standard iron solution, and

$W$  = weight in grams of sample in aliquot.

NOTE.—The above method for the determination of iron is to be used only when traces of iron are present. If larger quantities are found, the method described in Section 8 of the Standard Methods of Chemical Analysis of Limestone, Quicklime, and Hydrated Lime (A.S.T.M. Designation: C 25) of the American Society for Testing Materials<sup>3</sup> shall be used.

## PHYSICAL TESTS

### Sampling

12. The physical tests described in these methods shall be made on contain-

<sup>3</sup>1946 Book of A.S.T.M. Standards, Part II.

ers or parts of containers which have not been subjected to other tests or conditions which might affect the test results. A minimum of three containers will be required, one for the bulge test, one for the hot-cold cycle, and one for the tensile strength and acid absorption tests.

### TENSILE STRENGTH AND ELONGATION

#### Apparatus

13. (a) *Testing Machine*.—Tensile strength and elongation shall be determined on a power-driven apparatus of either the beam-weighing or pendulum type conforming to the following requirements:

(1) The applied tension as indicated by the dial or scale shall be accurate within 1 per cent.

(2) The indicator shall remain at the point of maximum load after rupture of the test specimen.

(3) The rate of travel of the power actuated grip shall be such that the average rate of load application shall be not more than 3.1 nor less than 2.9 lb. per sec. when measured with a non-extensible steel bar locked in the grips.

(4) Testing machines with a maximum capacity of 500 to 1500 lb. may be used to meet the conditions outlined above.

NOTE.—To establish a specification standard, it is advisable to specify the type of testing machine which is to be used to give the required tensile strength and elongation.

(b) *Grips*.—The grips shall be as described in Section 7 (b) of the Tentative Methods of Testing Hard Rubber Products (A.S.T.M. Designation: D 530).<sup>4</sup>

#### Calibration of Testing Machine

14. The testing machine shall be calibrated as described in Section 8 of the Tentative Methods of Testing Hard

Rubber Products (A.S.T.M. Designation: D 530).<sup>4</sup>

#### Test Specimen

15. (a) Test specimens shall be cut in the vertical direction by a band saw or similar means from the partitions of the container and shall be taken at least 1 in. from the top of the partition. The standard specimen shall conform in shape to Fig. 1, although a variation in

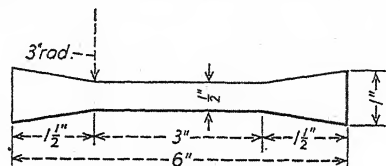


FIG. 1.—Standard Tension Test Specimen.

width of plus 0.005 or minus 0.025 in. shall be permitted. The variation in width of an individual specimen shall not be greater than 0.005 in. The specimens shall be tested without grinding the top and bottom faces; only the contour shall be cut to suitable template dimensions.

(b) Certain types of containers have partitions that are grooved in such a way that a standard tension test specimen cannot be cut from them. In such cases, the specimen used in testing hard rubber as shown in Fig. 2 or a similar

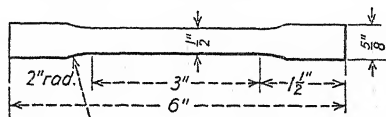


FIG. 2.—Alternative Tension Test Specimen.

specimen may be used. The size and shape of the specimen shall be included in the report.

NOTE.—The fact that results obtained from testing specimens which are not of the standard size and shape are not comparable should be noted and agreed upon by the purchaser and the manufacturer.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(c) Two parallel gage lines 3 in. apart, for use in determining elongation, shall be scratched on the reduced section of the specimen by means of a sharp-pointed instrument such as a pair of dividers. The lines shall be perpendicular to the longitudinal axis of the specimen, one on each side of the center and 1.5 in. therefrom. The marks shall be as fine as possible, consistent with legibility, and shall not be deep enough to cause a weakening of the specimen. If it becomes necessary to use a shorter test specimen (see Paragraph (b)), the gage lines shall be brought nearer together and the calculation for percentage elongation changed accordingly.

(d) The measurement, aging, and conditioning of the specimens and the temperature of test shall conform to the requirements prescribed in Sections 5 and 6 of the Tentative Methods of Testing Hard Rubber Products (A.S.T.M. Designation: D 530) of the American Society for Testing Materials.<sup>4</sup>

#### Number of Specimens

16. Five specimens from each sample shall be tested for determination of tensile strength and elongation. Results on specimens that break outside the straight reduced section or on specimens that are obviously defective shall be discarded and retests shall be made. Such retests may, however, be omitted if the test results conform to the specification requirements. For routine testing, three specimens have usually been found adequate.

#### Procedure

17. The test specimen shall be placed in the grips, using care to adjust it symmetrically in order that the tension shall be distributed uniformly over the cross-section. A tension load shall be applied to the specimen by the power-actuated grip, which shall travel at the

specified rate of speed until rupture takes place. During the application of the load the distance between the gage marks on the specimen shall be noted continuously by means of dividers so that the distance at the instant of rupture may be determined within 0.01 in. (Note). This value shall be recorded for use in calculating the ultimate elongation. After rupture of the specimen, the breaking load in pounds shall be read and recorded together with the original minimum width and thickness of the specimen so that the tensile strength may be calculated.

NOTE.—The measurement of elongation by means of an extensometer is not to be recommended unless the extensometer action is independent of the movement of the testing machine and unless the breaking strength is unaffected by reason of the attachment of the extensometer to the test specimen. For control purposes, such instruments may be found to be very acceptable.

#### Calculations

18. The tensile strength and elongation shall be calculated in accordance with Section 12 of the Tentative Methods of Testing Hard Rubber Products (A.S.T.M. Designation: D 530) of the American Society for Testing Materials.<sup>4</sup>

#### Report

19. The report shall include the following:

- (1) Date of test,
- (2) Temperature of test room,
- (3) Type of testing machine,
- (4) Description of sample and type of test specimen,
- (5) The observed and recorded data, and
- (6) The results calculated in accordance with Section 18.

#### BULGE TEST

##### Nature of Test

20. The purpose of this test is to measure the tendency of a battery



container to soften and bulge due to the heat generated when it is in service.

### Apparatus

21. The apparatus shall consist of the following:

(a) *Electrical Equipment*.—Means for passing an electric current through the cells of the container, when filled with an electrolyte.

(b) *Temperature Control*.—A control for maintaining constant temperature of the electrolyte during test.

(c) *Measuring Device*.—A device for

connections to provide for the passing of a continuous current. After the initial cooling when the electrolyte is placed in the container, the temperature shall be raised to  $160 \pm 2$  F. by means of an electric current and that temperature maintained by suitable electrical control for a period of 3 hr.

(c) After the container and acid have cooled to the temperature of the room, which should be maintained at  $75 \pm 5$  F., the width and length shall be measured again and the changes recorded.

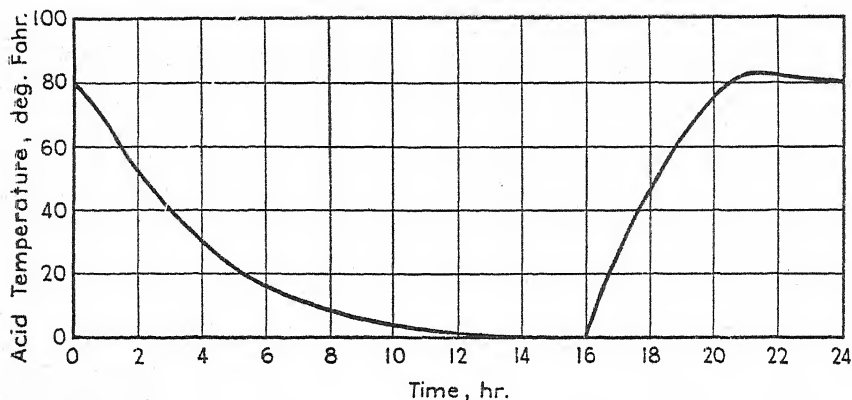


FIG. 3.—Hot-Cold Cycle for Sensitivity Test.

measuring the length and width of the container.

### Procedure

22. (a) The width and length of the battery container from the middle point of opposite panels shall be measured to the nearest  $\frac{1}{4}$  in. Each cell of the container shall be filled to a point approximately  $\frac{5}{8}$  in. from the top with water to which sufficient sulfuric acid has been added to give a specific gravity of approximately 1.01. The electrolyte shall be heated to a temperature of  $160 \pm 2$  F. before it is placed in the container.

(b) Carbon or lead electrodes shall be placed in the cells with the necessary

### Report

23. The report shall include the following:

(1) The original length, the length after test, and the increase in length, and

(2) The original width, the width after test, and the increase in width.

NOTE.—When two or more containers are tested at a time, a space of at least 2 in. shall be left between them to permit proper circulation of air.

### SENSITIVITY TO HOT-COLD CYCLES

#### Nature of Test

24. This test is designed to measure the tendency of a battery container to develop cracks as a result of abrupt and severe changes in temperature.

### Apparatus

25. The apparatus shall consist of the following:

(a) *Cold Chamber*.—A refrigerator or cold room of sufficient capacity to produce the temperatures required.

(b) *Hot Chamber*.—A hot chamber or room with heating means sufficient to produce the temperatures required.

### Procedure

26. (a) Each cell of the battery container shall be filled to a point 0.5 to 1.0 in. from the top with battery acid (sp. gr. 1.30 at 77 F.). Place the container in the refrigerator, which shall be maintained at a temperature of  $0 \pm 2$  F. for a period of 16 hr. The capacity of the refrigerator shall be sufficient to reduce the temperature of the acid in the middle cell of the container to  $0 \pm 2$  F. in 13 to 15 hr.

(b) Upon withdrawal of the container from the refrigerator, it shall be placed directly into the hot chamber. The hot chamber shall have a temperature between 110 and 120 F. and shall be of such capacity as to cause the acid in the middle cell of the container to reach  $83 \pm 2$  F. in 5 hr.

(c) After 5 hr. in the hot chamber the container shall be withdrawn and allowed to rest 3 hr. at room temperature, which will complete the first cycle of 24 hr. Figure 3 shows graphically the hot-cold cycle required for this test. Not only initial and final temperatures, but also the intermediate temperatures, shall be approximated.

(d) Examination of the container shall be made to note the development of any cracks and leaks, and the cycle shall be repeated until such time as cracks may develop, or until the required number of cycles have been completed.

NOTE.—Precautions must be taken, when several containers are to be tested at one time, to enable each container to reach the required temperature levels by proper spacing of the

containers and regulation of the refrigerator and hot chamber. Circulation of the air in both the refrigerator and the hot chamber are necessary to produce uniform conditions.

### Report

27. The report shall include the following:

(1) Record of the temperatures attained in each cycle, and

(2) Number of cycles before failure occurs.

## ACID ABSORPTION

### Nature of Test

28. This test is intended to measure the penetration of battery acid into the partitions of a battery container at an elevated temperature.

### Test Specimen

29. (a) The test specimen shall be 2 in. square and of the thickness of the partition from which it is cut. The specimen shall be taken at least 1 in. from the top of the partition, and shall not contain any irregular flow marks.

(b) Since the rate of penetration of acid through the cut edges is different from the rate through the sides, the edges may be sealed to overcome this difficulty. A high melting mastic or other acidproof adhesive may be used for this purpose.

### Number of Tests

30. Two test specimens shall be tested and the average values used in determining the acid absorption and penetration.

### Procedure

31. (a) Weigh the test specimen and submerge it in  $H_2SO_4$  (sp. gr. 1.30 at 77 F.) in a jar or glass container. When several test specimens are to be placed in the same container, glass triangles or other suitable means shall be used to separate the specimens from

each other. Fresh acid shall be used for each new test.

(b) Cover the jar to avoid evaporation and concentration of the acid; then place it in an oven maintained at  $150 \pm 2$  F., for a period of 28 days. At the conclusion of this period, withdraw the specimen and wash it in running tap water for approximately 1 min. to remove the acid from the surface of the specimen. Wipe the surface with a cotton cloth to take up residual water, and weigh the specimen immediately.

(c) After the test specimen is weighed, cut it through the center into two pieces, each about 2 in. in length and 1 in. in width. Polish the freshly cut edge with benzene or other suitable solvent, and measure the average penetration of the acid to the nearest  $\frac{1}{16}$  in. In determining the average penetration only the area within 0.5 in. of the middle point shall be noted.

(d) *Alternative Method.*—If the 28-day period for the test is impracticable, a shorter period of 7 days at  $150 \pm 2$  F. may be substituted. The procedure shall otherwise be carried out as described in Paragraphs (a) to (c) (Note).

NOTE.—The 7-day period of immersion in acid usually results in less penetration of the acid than in the preferred test period of 28 days.

### Report

32. The report shall include the following:

(1) Increase in weight of the test specimen, expressed in grams (indicate whether or not the edges are sealed),

(2) Penetration of the acid into the interior of the test specimen, expressed in sixty-fourths of an inch,

(3) Time in acid, and

(4) Temperature of acid.

# *Tentative Methods of* TESTING CELLULAR RUBBER PRODUCTS<sup>1</sup>



**A.S.T.M. Designation: D 552 - 46a T**

*(Revision of Methods D 552 - 46 T)*

ISSUED 1939; REVISED, 1941, 1943, 1944, JANUARY 1946, JUNE 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. (a) These methods cover procedures for testing cellular rubber products in which the base material used in manufacture may be natural rubber, reclaimed rubber, synthetic rubber or rubber-like materials, alone or in combination. Cellular rubbers may be vulcanized either to soft or to hard (ebonite) rubber.

(b) The cellular rubber products to which these methods are applicable include sponge rubbers, latex foam rubbers, and expanded rubbers. The methods included are not all applicable to every class of cellular rubbers nor do they necessarily include every test applicable to a particular class of cellular rubber. References to these methods for testing cellular rubber products should specifically state the particular test or tests desired.

## General Methods

2. (a) Except as otherwise specified in these methods of testing cellular rubbers,

the following methods of test of the American Society for Testing Materials, applicable in general to vulcanized rubber, shall be complied with as required and are hereby made a part of these test methods:

*General Physical Test Requirements.*—Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15).<sup>3</sup>

*Aging Test.*—Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (A.S.T.M. Designation: D 572),<sup>3</sup> Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573),<sup>3</sup> and Standard Method of Air Pressure Heat Test of Vulcanized Rubber (A.S.T.M. Designation: D 454).<sup>3</sup>

*Compression Set.*—Tentative Methods of Test for Compression Set of Vulcanized Rubber (A.S.T.M. Designation: D 395).<sup>3</sup>

*Chemical Analysis.*—Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297).<sup>3</sup>

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Latest revisions accepted by the Administrative Committee on Standards, January 10, 1946, and by the Society at annual meeting, June, 1946

<sup>3</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

**Low-Temperature Brittleness.**—Tentative Method of Test for Low-Temperature Brittleness of Rubber and Rubber-Like Materials (A.S.T.M. Designation: D 736).<sup>3</sup>

**Compression - Deflection.**—Standard Methods of Test for Compression-Deflection Characteristics of Vulcanized Rubber (A.S.T.M. Designation: D 575).<sup>3</sup>

**Fluid Immersion.**—Tentative Methods of Tests for Changes in Properties of Rubber and Rubber-Like Materials in Liquids (A.S.T.M. Designation: D 471).<sup>3</sup>

(b) In case of conflict between provisions of the above methods and the procedures herein specifically described for cellular rubbers, the latter shall take precedence. In case of conflict between the procedures herein described for cellular rubbers and the methods of a particular specification or for a particular cellular rubber product, the latter shall take precedence.

### Sampling

3. (a) When possible, the completed manufactured product shall be used for the tests specified. Representative samples of the lot being examined shall be selected at random as required.

(b) When it is necessary or advisable to obtain test specimens from the article, as in those cases where the entire sample is not required or adaptable for testing, the method of cutting and the exact position from which specimens are to be taken shall be specified. The apparent density and the state of cure may vary in different parts of the finished product, more especially if the article is of complicated shape or of varying thickness, and these factors affect the physical properties of the specimens. Also, the apparent density is affected by the number of cut surfaces as opposed to the number of skin-covered surfaces on the test specimen.

(c) When the finished product does not

lend itself to testing or to the taking of test specimens because of complicated shape, small size, metal or fabric inserts, solid covers, adhesion to metal, or other reasons, standard test slabs shall be prepared. Standard test slab specimens shall also be used for arbitration purposes in the event of controversy concerning test results obtained from samples of commercial articles.

### Standard Test Specimens

4. Standard test specimens shall be disks 1.129 in. in diameter. The specimens may be cut with a revolving die<sup>4</sup>

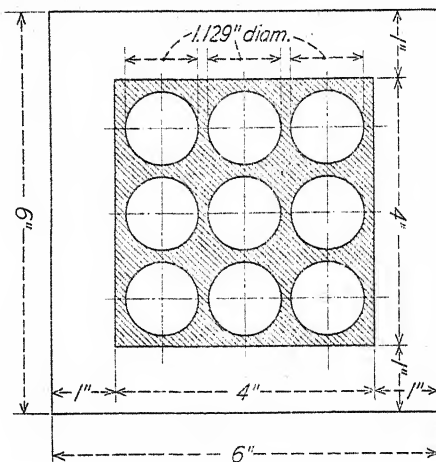


FIG. 1.—Location From Which Standard Test Specimens Are to be Cut When Testing Standard Test Slabs or Commercial Flat Sheets.

using a soap solution as a lubricant. If a lubricant is used the specimens shall be thoroughly dried before proceeding with the testing. In some cases it may be necessary to freeze the cellular rubber to obtain parallel cut edges. When cut from standard test slabs they shall be cut from the center area as shown in Fig. 1.

<sup>4</sup> A satisfactory die and its method of application are described in Section 4 of the Standard Methods of Test for Compression-Deflection Characteristics of Vulcanized Rubber (A.S.T.M. Designation: D 575), see p. 299.

The thickness shall be measured using the dial micrometer gage described in Section 6 (b).

### Standard Test Slabs

5. (a) Standard test slabs of all types of cellular rubber shall be pieces 6-in. square and  $\frac{1}{2}$  in. in thickness made from the same compound and having the same apparent density and state of cure as the product they represent. In all cases the surface skin shall be left intact on both top and bottom faces. Standard test slabs shall be prepared either by cutting them from commercial flat sheets of the specified thickness or, in the case of sponge rubbers, by the special procedure described in the following Paragraph (b).

(b) When specially prepared standard test slabs of sponge rubber are required, they shall be made using the frame shown in Fig. 2 together with top and

cavities. The thickness of the square sheets shall be such as to give the required apparent density when the material is blown during cure to fill the molding cavities. The squares of stock shall be dusted with talc and the excess brushed off to avoid pitting. They shall then be placed in the frame, and fabric sheeting shall be applied on the top and bottom between the frame and the plates to allow venting of gases produced during the cure. This fabric shall be a commercial sheeting weighing approximately 4 oz. per sq. yd., having approximately 70 ends per in. and 60 picks per in. The specimens shall be vulcanized in a platen press under conditions of time and temperature chosen to produce the same state of cure in the standard slabs as in the finished products they represent.

### Measurements of Finished Products

6. (a) The length and width shall be measured with a steel scale or tape. Care shall be taken not to distort the cellular rubber.

(b) Thicknesses up to and including 1 in. shall be measured using a dial-type gage<sup>5</sup> having a maximum stem and foot weight of 25 g. and a foot  $1\frac{1}{4}$  in. in diameter. Thicknesses over 1 in. shall be measured using a sliding caliper gage or as specified in Paragraph (a). When a sliding caliper gage is employed, the gage setting shall be made with the gage out of contact with the cellular rubber. The sample shall be passed through the previously set gage and the proper setting shall be the one when the measured faces of the gage contact the surfaces of the article without compressing it.

(c) The type of steel scale, tape or dial gage and the graduations employed shall be so chosen as to give accurate measurements according to the specified permissible variations.

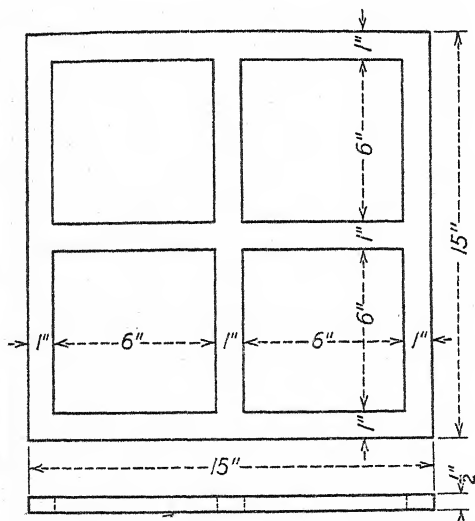


FIG. 2.—Four-Cavity Frame for Standard Test Slabs of Cellular Rubbers.

bottom plates each approximately 0.50 in. in thickness. The frame and plates shall be made of aluminum or steel. The stock shall be in sheet form cut into squares slightly smaller than the frame

<sup>5</sup> A gage similar to Federal Products Co. No. D-81-S is satisfactory.

(d) Results reported shall be the average of a minimum of three measurements.

#### CHEMICAL ANALYSIS

##### Procedure

7. The chemical analysis of cellular rubber products shall be made in accordance with the complete procedure as described in the Tentative Methods of Chemical Analysis of Rubber Products (A.S.T.M. Designation: D 297).<sup>3</sup>

#### APPARENT DENSITY

##### Procedure

8. (a) The apparent density shall be calculated by dividing the weight of the cellular rubber by its volume. In the case of cored stock, the volume as measured shall include the volume of the core holes. The apparent density shall be expressed in ounces per cubic inch.

(b) All weighings shall be made to an accuracy of 0.25 per cent.

(c) The volume of regularly shaped products or specimens shall be obtained from measurements of length, width, and thickness.

(d) The volume of irregularly shaped products or specimens, which cannot be measured by ordinary means, shall be obtained by the displacement method, as follows: The test specimen shall be placed in a graduated container of slightly larger dimensions than the specimen and completely surrounded by 10 to 14-mesh, free-flowing seed, such as rapeseed, so that the specimen is covered to a depth of 1 in. Test specimens containing core holes shall have the holes sealed off with cellophane or similar suitable material before being placed in the container. The seed shall be added to the graduated container under controlled conditions by use of a glass powder funnel having a top diameter of 100 mm. and a stem diameter of 18 mm. The bottom of the funnel shall be placed

6 in. above the top of the specimen. The apparent volume of the seed and the specimen shall be recorded. After removing the test specimen, the apparent volume of the seed shall be recorded, and the volume of cellular rubber calculated as follows:

$$V_1 = (V_1 + V_2) - V_2$$

where:

$V_1$  = volume of cellular rubber, and

$V_2$  = apparent volume of seed.

(e) A minimum of three tests shall be made and the average value reported.

#### ACCELERATED AGING TESTS

##### Procedure

9. Either the oxygen-pressure-chamber aging test, the air-oven aging test, or the air-pressure heat test as described in the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oxygen-Pressure Method (A.S.T.M. Designation: D 572),<sup>3</sup> by the Oven Method (A.S.T.M. Designation: D 573),<sup>3</sup> or by the Air Pressure Heat Test (A.S.T.M. Designation: D 454),<sup>3</sup> respectively, may be used for cellular rubbers, as specified, except that in the air-pressure heat test an air pressure of  $60 \pm 2$  psi. shall be used in place of the  $80 \pm 2$  psi. prescribed in Method D 454. Deterioration may be expressed as percentage change of compression-deflection values or the results may be determined by visual observation. No relation between accelerated aging tests and natural aging is given or implied.

#### COMPRESSION-DEFLECTION TESTS

##### Test Specimens

10. Standard test specimens shall be used for this test. They shall be cut so that opposite edges are parallel, either from the finished product in a manner agreed upon by the parties concerned or, as shown in Fig. 1, from standard test slabs or from commercial flat sheets.



The thickness of the test specimens may vary, but shall be measured and stated in the report.

NOTE.—The compression-deflection test is ordinarily made on sponge rubbers and expanded rubbers. The indentation test (Sections 16 to 20) is usually preferred on latex foam rubbers.

### Procedure

11. (a) Before starting the test, the specimen of cellular rubber shall be compressed 25 per cent of its free height by applying and releasing sufficient load three times in succession after which the test shall be made immediately. Cellular rubbers less than  $\frac{1}{4}$  in. in thickness shall be tested by plying up the proper number of plies to obtain a thickness as near  $\frac{1}{2}$  in. as possible.

(b) The standard test specimen shall be compressed between parallel metal plates by applying a load sufficient to reduce the thickness by 25 per cent of its original value. The bottom and top plates shall be at least 1.5 in. in diameter. The load shall be applied as a dead weight. The reading shall be taken immediately after the load has been applied. Any arrangement of applying the load so that the two plates are kept parallel is satisfactory.

### Report

12. The unit load required expressed in pounds per square inch shall be reported as the result of the compression-deflection test.

### COMPRESSION SET UNDER CONSTANT DEFLECTION

#### Test Specimens

13. Standard test specimens shall be used for this test. They shall be cut so that opposite edges are parallel, either from the finished product in a manner agreed upon by the parties concerned or, as shown in Fig. 1, from standard test

slabs or from commercial flat sheets. The thickness of the test specimens may vary, but shall be measured and stated in the report.

### Procedure

14. The apparatus and procedure shall be the same as prescribed in Method B of the Tentative Methods of Test for Compression Set of Vulcanized Rubber (A.S.T.M. Designation: D 395),<sup>3</sup> except as follows: Test specimens shall be compressed 50 per cent of their original thickness. The load shall be released at the end of the test period and the thickness measured after 30 min. rest at room temperature. Thickness measurements shall be made as described in Section 6. The temperature of the test shall be 158 F. The time of the test shall be as specified.

### Calculations

15. The percentage compression set shall be calculated as follows:

$$\text{Compression set, per cent} = \frac{h_1 - h_2}{h_1} \times 100$$

where:

$h_1$  = original height, and

$h_2$  = final height.

### INDENTATION TEST

#### Scope

16. This test is applicable to the testing of latex foam rubbers. It consists of measuring the load necessary to produce a 25 per cent indentation in the cellular rubber product.

#### Apparatus

17. An apparatus having a flat circular indenter foot 50 sq. in. in area, connected to a load measuring device, and mounted in such a manner that the product or specimen can be deflected at a specified rate, shall be used for this test. The apparatus shall be arranged to sup-

port the specimen on a level horizontal plate which is perforated with 0.25-in. holes on 0.75-in. centers to allow for rapid escape of air during the test.

#### Test Specimens

18. The test specimen shall consist of the entire product sample or a suitable portion of it, except that in no case shall the surface for indentation have dimensions less than 12 by 12 in.

#### Procedure

19. (a) The test specimen shall be placed in position on the supporting plate of the apparatus. In case the product has one side cored or honeycombed, this face shall rest on the perforated plate. The specimen position shall be such that the indentation will be made at the center of all articles.

(b) The indenter foot shall be brought into contact with the specimen, and the original height shall be determined after applying a total preload of 1 lb. The product shall then be compressed 25 per cent of this original height and the final load in pounds observed. The result obtained in this test is influenced by temperature and humidity conditions, and tests which are to be compared shall be conducted under substantially the same conditions. In all cases, the actual temperature and humidity during the test shall be reported.

(c) In cases of dispute, the compression readings shall be performed at a temperature of  $77 \pm 3.6$  F. and in an atmosphere having a relative humidity of  $60 \pm 5$  per cent. The product shall be conditioned undeflected and undistorted at this temperature and humidity for at least 12 hr. before being tested. Ordinarily only one test shall be made, but in cases of dispute the result shall be expressed as the average of a minimum of three tests.

#### Calculations

20. Readings obtained on the indenter foot shall be divided by 50 and the result expressed as pounds per square inch.

#### FLEXING TEST

##### Scope

21. The flexing test is applicable to the testing of latex foam rubbers. It consists of subjecting the test specimen to repeated compression and noting the effect on the cellular structure.

##### Test Specimens

22. The test specimen shall consist of the entire product sample or a suitable portion of it, except that in no case shall the loading surfaces have dimensions less than 12 by 12 in.

##### Procedure

23. The test specimen shall be placed centrally on the stationary plate of the flexing machine. The flexing plates of the machine shall be at least  $\frac{1}{4}$  in. larger on each side than the specimen being tested. In case the product has one side cored or honeycombed, that face shall rest on the perforated plate. The plate positions shall then be adjusted so that the loading plate is brought just into contact with any part of the specimen. The distance between the two plates shall then be taken as the original thickness of the specimen. For products having an indentation value of less than 1.72 psi. the amplitude of compression and decompression shall be 50 per cent of the original thickness. For products having an indentation value of 1.72 psi., or higher, the amplitude shall be 25 per cent of the original thickness. The machine shall then be started reciprocating at 60 cycles per minute and the total number of flexures recorded continuously by means of a counter. Failure of the specimen is evidenced by physical

breakdown of the cellular structure as determined by visual examination and comparison with unflexed specimens. Specimens may be evaluated as agreed upon by the manufacturer and the purchaser either by flexing them a specified number of times and comparing the degree of resultant physical breakdown or by flexing them to failure and comparing the number of flexures required. The compression set shall be measured 30 min. after completion of the test.

### Procedure

25. When testing with petroleum base lubricating oils, it is recommended in the interest of standardization that the test be conducted in one of the oils prescribed in Table I which has an aniline point nearest that of the oil with which the cellular rubber is expected to come in contact in service. The aniline point of a petroleum oil appears to characterize the swelling action of that oil on synthetic cellular rubber compounds. In

TABLE I.—PETROLEUM-BASE LUBRICATING OILS.\*

	Petroleum-Base Oils			A.S.T.M. Designation <sup>a</sup>
	No. 1, Low Swelling, Aniline Point 250 F.	No. 2, Medium Swelling, Aniline Point 200 F.	No. 3, High Swelling, Aniline Point 160 F.	
Saybolt Universal Viscosity, sec.....	98 ± 5 <sup>b</sup>	100 ± 5 <sup>b</sup>	155 ± 5 <sup>c</sup>	D 88
Aniline point.....	123.9 ± 1 C. (255 ± 1.8 F.)	95 ± 3 C. (199.4 ± 5.4 F.)	69.5 ± 1.5 C. (157.1 ± 1.8 F.)	D 611
Flash point, deg. Fahr.....	470 min.	475 min.	330 ± 5	D 92

\* These designations refer to the following methods of the American Society for Testing Materials:

Standard Method of Test for Viscosity by Means of the Saybolt Viscosimeter (A.S.T.M. Designation: D 88),<sup>7</sup>

Tentative Method of Test for Aniline Point and Mixed Aniline Point of Petroleum Products (A.S.T.M. Designation: D 611),<sup>7</sup> and

Standard Method of Test for Flash and Fire Points by Means of Open Cup (A.S.T.M. Designation: D 92).<sup>7</sup>

<sup>b</sup> Measured at 210 F.

<sup>c</sup> Measured at 100 F.

## OIL IMMERSION TEST

### Test Specimens

24. Standard test specimens approximately  $\frac{1}{2}$  in. in thickness shall be used for this test. The diameter and thickness shall be measured before and after immersion in the specified petroleum-base oil for 22 hr. at 158 F. and the percentage change in volume calculated. Three specimens shall be run on each test and the average of the three values reported.

general, as the aniline point becomes lower the swelling action of the oil becomes more severe. The oils shown in Table I cover a range of aniline points commonly found in lubricating oils.

### LOW-TEMPERATURE TEST

#### Apparatus

26. The apparatus shall consist of two parallel plates at least 1.5 in. in diameter, one of which is movable and the other one stationary, a means of applying a load and a means of accurately measuring the distance between the parallel plates.

#### Test Specimens

27. Standard test specimens shall be used for this test. The thickness may be different from that of the standard specimen, but it shall be measured and stated in the report.

<sup>6</sup> The following suppliers state that their products meet the requirements for these petroleum-base oils when ordered as A.S.T.M. oils as follows:

A.S.T.M. Oil No. 1 may be ordered as such from Stanco Distributors, Inc., Chemical Products Dept., Attention Mr. A. B. Boehm, 26 Broadway, New York 4, N. Y.

A.S.T.M. Oil No. 2 may be ordered as Sun XX Heavy Cable from Sun Oil Co., Philadelphia, Pa.

A.S.T.M. No. 3 may be ordered as such from Sun Oil Co., Attention Mr. J. B. Hill, Marcus Hook, Pa.

<sup>7</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

**Procedure**

28. The compression-deflection of the specimen shall first be measured at room temperature and the load in pounds per square inch necessary to obtain a 25 per cent deflection recorded. The specimen shall then be placed in the cold box for 5 hr. at a temperature of  $-40^{\circ}\text{F.}$  at the end of which time the previously determined load shall be applied as *rapidly* as possible while the specimens are still in the cold box and the deflection recorded immediately.

**Calculation**

29. The percentage change in deflection shall be calculated as follows:

$$C = \frac{D - E}{D} \times 100$$

where:

$C$  = percentage change in deflection,

$D$  = deflection at room temperature,  
and

$E$  = deflection at  $-40^{\circ}\text{F.}$

**WATER ABSORPTION TEST****Scope**

30. The water absorption test is applicable to expanded rubbers (closed cell type). It should not be used on sponge rubbers or latex foam rubbers (open cell type) unless they are completely encased in an added skin.

**Test Specimens**

31. Standard test specimens approximately  $\frac{1}{2}$  in. in thickness shall be used for this test. Test specimens shall have the natural skin on the top and bottom surfaces of the disks.

**Procedure**

32. Specimens shall be submerged in distilled water at room temperature ( $65$  to  $90^{\circ}\text{F.}$ ) 6 in. below the surface of the water for 22 hr. and then dried off with an air blower and the change in weight calculated.

# *Tentative Methods of*

## TESTING RUBBER ADHESIVES<sup>1</sup>



A.S.T.M. Designation: D 816 - 46 T

ISSUED, 1944; REVISED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods are intended for use in testing the properties of adhesives which may be applied in plastic or fluid form and which are manufactured from natural rubber, reclaimed rubber, synthetic elastomers, or combinations of these materials. They are not all to be considered as applicable to a particular type of adhesive nor do they necessarily include every test which may be applicable to a particular type. They do, however, provide procedures in standard form for evaluating the more important properties of the usual adhesives ordinarily classed as rubber cements.

### Classes of Tests

2. The tests described in these methods may be classified in two groups, the first including those procedures in which the adhesive is applied to specimens of materials to be bonded after which the quality of the bond is evaluated, and the second including those procedures applicable to the adhesive itself without consideration of the bonding properties.

The tests in class 1 are those for adhesion strength, bonding range, softening point, and cold flow. Those in class 2 comprise determinations of viscosity, stability, cold brittleness, weight per gallon, and plastic deformation (for heavy doughs or putties).

### Materials for Bonded Specimens

3. The following materials shall be used as specified for the preparation of specimens bonded with the test cement for tests of class 1 and shall be reported with the result of the test:

Aluminum sheet, clad material, gage 0.064 in.

Standard rubber test slabs.<sup>a</sup>

Cotton duck (30.31 oz. per sq. yd., medium texture, A.S.T.M. Specifications D 230).<sup>b</sup>

Coated fabrics of which the fabric strength and the adhesion of coating to fabric exceed the strength of the adhesive bond.

Rolled sheet or machined steel, S.A.E. No. 1020, sand blasted or sanded with 240 grit abrasive.<sup>c</sup>

Special materials such as leather, cork, felt, etc.

<sup>a</sup> See Section 4 of the Standard Methods of Sample Preparation for Physical Testing of Rubber Products (A.S.T.M. Designation: D 15).<sup>3</sup>

<sup>b</sup> Standard Specifications for Numbered Cotton Duck and Army Duck (A.S.T.M. Designation: D 230).<sup>4</sup>

<sup>c</sup> 1946 S.A.E. Handbook, p. 300.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-11 on Rubber and Rubber-Like Materials.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

NOTE.—In reporting tests using aluminum sheet, the alloy and type of cladding shall be stated. Care shall also be taken when rubber test slabs are used for bonded specimens to report the compound actually employed. In the case of coated fabrics, the exact nature of the coating shall be stated. Any special materials shall be completely identified and described in the report.

### Sampling

4. Before sampling, the cement to be tested shall be thoroughly agitated to uniform consistency throughout, after which a sample shall be taken of sufficient quantity for performance of the tests required. The sample shall be kept in a tightly closed container to prevent evaporation and shall be agitated or stirred to uniform consistency immediately before test or use in preparation of test specimens.

### Test Conditions

5. The tests of adhesives shall be conducted at a temperature of  $77 \pm 5$  F. and relative humidity of  $50 \pm 5$  per cent. All material and equipment shall be held at these conditions prior to test for a sufficient time to assure equilibrium.

## ADHESION TESTS

### Types of Adhesion Test

6. The adhesion strength of a cement used to bond two materials is measured by determining the force required to produce separation by failure of the adhesive. This force may be applied as (1) a direct tension pull in the direction normal to the adhered surfaces, (2) a shearing force in the same plane as the surfaces, or (3) a stripping force which produces progressive separation over the adhered surfaces. It is obvious that the stripping procedure can be used only when at least one of the adhered materials is flexible. Methods, designated A, B, and C, are given for preparing and testing bonded specimens by each of these types of adhesion test. The adhesion

test may also be applied, when specified, to bonded specimens which have been subjected to deteriorating influences such as accelerated aging or action of oils or solvents after their preparation and before separation but in this case full details concerning such treatment shall accompany the statement of the result of the adhesion test.

### *Method A. Adhesion Strength in Tension*

#### Preparation of Test Specimens

7. (a) For testing cements for use in vulcanized applications, the test specimens shall be those specified in Sections 3 and 4 of the Standard Method of Test for Adhesion of Vulcanized Rubber to Metal (A.S.T.M. Designation: D 429),<sup>3</sup> except that after buffing or grinding the surfaces to be adhered, and washing them free from dirt or oil using carbon tetrachloride, they shall be coated with a uniform film of the test adhesive as near as possible 0.001 in. in thickness.

(b) For testing cements for use in unvulcanized applications, test specimens similar to those specified in Paragraph (a) shall be prepared except that the rubber cylinders used in their preparation shall be cut from vulcanized rubber and the assembly of the specimen shall be made without vulcanization in a mold. To accomplish this, the circular faces of the rubber cylinders shall be buffed smoothly and evenly to parallel planes and shall be coated with a uniform film of the test cement as near as possible 0.001 in. in thickness. With thin cements, the coating may be done with a brush, applying successive coats and allowing sufficient time between coats for evaporation of the solvent. The faces of the metal plates shall be freshly sand-blasted or ground to a dull finish with fine emery and washed free of dust and oil with carbon tetrachloride. The

metal faces shall also be coated with the test cement as in the case of the rubber cylinders. When the cement films have dried to the point where there is still aggressive tackiness but no tendency for the film to lift when touched with a finger, each rubber cylinder shall be bonded firmly by hand between the cemented faces of two metal plates and the assembly shall be clamped in a suitable loading fixture under a compressive load of 10 lb. for at least 24 hr.

### Procedure

8. The prepared specimens shall be tested as described in Section 6 of A.S. T.M. Method D 429. Care shall be taken to use a tension testing machine of capacity and sensitivity suitable for

### Method B. Adhesion Strength in Shear

#### Types of Test Specimens

9. Specimens for shear tests shall be of two types, namely, Type 1 lap specimens, and Type 2 sandwich specimens as shown in Fig. 1. Lap specimens shall be used when the adhesive is to be tested for bonding materials which are both extensible such as rubber, felt, or cork, or both relatively nonextensible like metal, cloth, and leather. Sandwich type specimens shall be used when one of the bonded materials is extensible and the other nonextensible. In both types of specimens, two strips of the material 1 in. in width and 5 in. in length shall be bonded over an area of 1 sq. in. with the free ends extending parallel in opposite

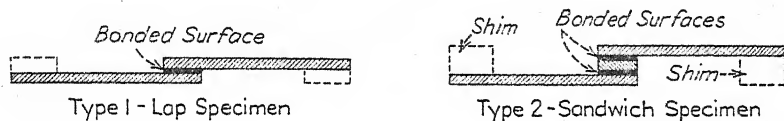


FIG. 1.—Test Specimens for Adhesion Strength in Shear.

loads of the magnitude anticipated with the particular adhesive being tested. The adhesion strength shall be expressed in pounds per square inch calculated by dividing the tension load causing failure by the original area of the adhered surface which separates. With the standard test specimens the value is the load at failure divided by two. Two specimens shall be tested and the higher of the results shall be reported as the adhesion strength in tension provided they check within 10 per cent. Otherwise, additional specimens shall be tested until results are obtained consisting of the high value and one which checks this within 10 per cent. If no check of the high value is obtained when six specimens have been tested the average of all six results may be reported as the average adhesion strength in tension.

directions to permit clamping in a testing machine. In the case of the lap-type specimens the two strips shall be bonded directly to each other by means of the adhesive being tested but in the case of the sandwich-type specimen, a 1-in. square block of extensible material having a thickness of not more than  $\frac{1}{8}$  in. shall be bonded between two nonextensible strips by means of the test adhesive.

#### Preparation of Test Specimens

10. The faces of the materials which are to be bonded by the test adhesive shall be buffed by hand using a wire brush, fine emery cloth or similar suitable abrasive to assure a slightly roughened surface which shall then be washed free of dirt and oil by means of oil-free carbon tetrachloride. When thoroughly



dry and without touching the surfaces after washing, the test adhesive shall be applied by brushing, spraying or spreading to form a film having a thickness of 0.001 in. as near as possible.

NOTE.—With cements of sufficiently heavy consistency to allow their use, the volumetric or dead weight cement spreaders shown in Figs. 2 and 3 may be found convenient for applying the adhesive in preparing the required specimens. With thin cements applied by means of brushing, several coats may be necessary in which case adequate drying time to permit evaporation of the solvent should be allowed between coats. Three such coats will usually be found sufficient.

### Procedure

11. After the required aging period, the prepared specimen shall be clamped in the grips of a tension testing machine conforming to the requirements prescribed in Section 6 of the Standard Methods of Test for Adhesion of Vulcanized Rubber (Friction Test) (A.S. T.M. Designation: D 413),<sup>3</sup> using shims in the grips as indicated in Fig. 1 so that the applied force shall be in the plane of the cemented area. Care shall be taken to be certain that the specimen is properly centered and is straight in the grips.

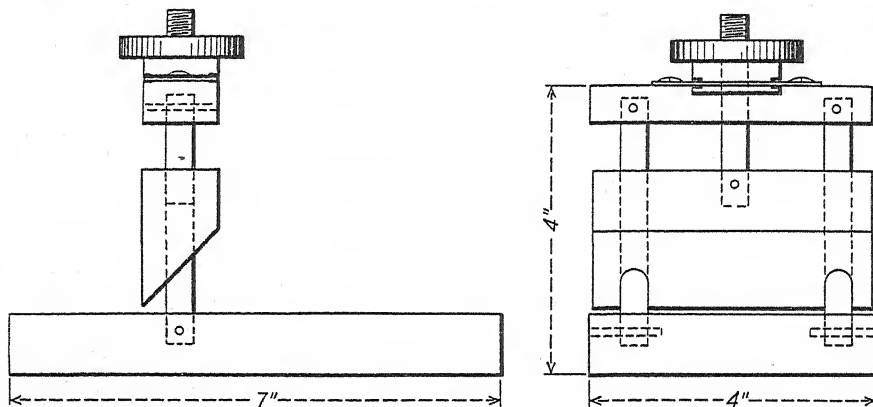


Fig. 2.—Volumetric Cement Spreader.

When the cement film has dried to the point where there is still aggressive tackiness but no tendency for the film to lift when touched with a finger, the surfaces to be bonded shall be aligned carefully so that 1 sq. in. shall be adhered and pressed together firmly by hand. The specimens, resting on a smooth hard base, shall then be rolled with five complete strokes using a metal roller 1 in. in diameter applied with a hand pressure of approximately 20 lb. The specimens shall then be allowed to age for at least 24 hr. under the standard test conditions of temperature and humidity (Section 5) without any applied pressure.

When testing type 1 specimens made of extensible materials, provision shall be made to maintain the strip during the test approximately in the plane of the clamps. This may be done by holding the specimen against a plate attached to the stationary clamp. The clamps shall be separated at the rate of 2 in. per min. and the load causing separation of the specimen shall be recorded in pounds per square inch of the adhered surface which separates. (*Caution:* When shims or plates are added to the clamps, the testing machine must be adjusted for zero load with these additions or their weight must be deducted from the indicated

reading.) Two specimens in each required combination shall be tested and the higher of the results shall be reported as the adhesion strength in shear provided they check within 10 per cent. Otherwise, additional specimens shall

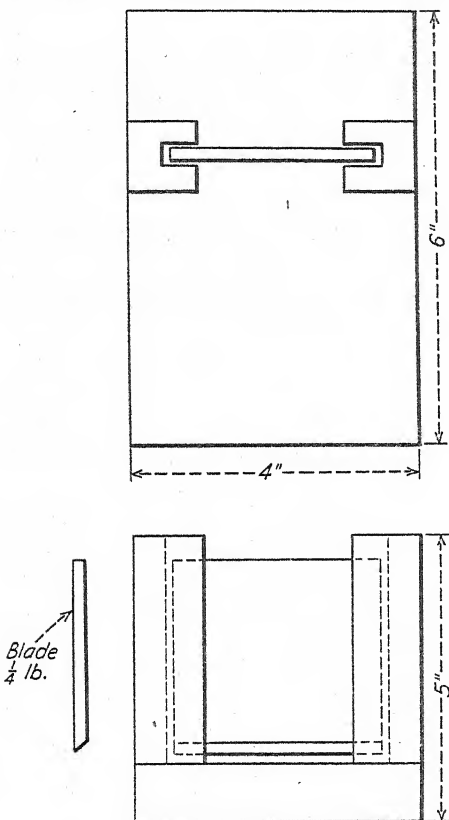


FIG. 3.—Dead Weight Cement Spreader.

be tested until results are obtained consisting of the high value and one which checks this within 10 per cent. If no check of the high value is obtained when six specimens have been tested, the average of all six results may be reported as the *average* adhesion strength in shear.

#### *Method C. Adhesion Strength by Stripping Method (Friction Test)*

##### **Preparation of Test Specimens**

12. A strip at least 6 in. in length by

1 in. in width of flexible material shall be bonded by means of the test cement except for 2 in. at one end to a strip 12 in. in length by 2 in. in width of the other material which is to be adhered. The center lines of the two strips shall be superimposed. The surface to be bonded shall be properly prepared, for example, by buffing, washing with carbon tetrachloride, drying and coating with a film of the test cement 0.001 in. in thickness (Note), uniting, rolling down, and aging as specified in Section 10 for shear test specimens.

NOTE.—In the case of porous material, the thickness of the cement film shall not be specified.

##### **Procedure**

13. The test specimens shall be placed in a tension testing machine by clamping the free end of the 2-in. wide strip in one grip and turning back the free end of the flexible 1-in. wide strip and clamping it in the other grip. The testing machine and the procedure used shall conform to that specified under the machine method for strip specimens in Sections 6 and 7 of A.S.T.M. Method D 413. The autographic chart obtained constitutes the report of the test but the value of the adhesion shall be determined by drawing on the chart the best average line between the maximum and minimum load values. The load so indicated, expressed in pounds per inch of width for separation at 1 in. per min., shall be considered as the adhesion strength by the stripping method. For the evaluation of each cement sample, two samples shall be prepared and tested and the higher average load obtained shall be reported as the adhesion strength of the sample.

##### **BONDING RANGE**

##### **Type of Test**

14. The test for bonding range is intended for use in determining the most

suitable drying period for use with the test cement after application and before bonding the materials in order to obtain the maximum initial bond strength. A series of type 2 shear adhesion test specimens (Fig. 1) is prepared using the test cement and allowing it to dry for different known periods before uniting the materials. These specimens are cemented, dried, bonded, and tested for adhesion strength in shear according to a definite time schedule. The drying period which results in the highest shear adhesion value is the optimum bonding range for the cement being tested.

#### Preparation of Test Specimens

15. The test specimens shall be type 2 shear adhesion test specimens each made from two strips of aluminum alloy 1 in. in width by 5 in. in length bonded by the test cement to a block of heavily compounded rubber 1-in. square by  $\frac{1}{8}$  in. in thickness having a durometer hardness of 75 to 85. The specimens shall be prepared in duplicate as specified in Section 10 except that the application of the cement shall be so scheduled that successive pairs of specimens may be dried 1, 3, 5, 10 and 20 min. before bonding and each tested exactly 3 min. after making the bond. After the first 20 min. of drying time, additional intervals of 20 min. to a maximum of 2 hr. shall be used.

#### Procedure

16. The test specimens prepared according to the prearranged schedule shall be tested as specified in Section 11 three minutes after making the bond and rolling them. The force in pounds required to separate the rubber from the metal shall be recorded for each specimen and the maximum value selected. The drying time used for cementing that specimen shall be reported as the optimum bonding range. If the maximum value is obtained with a specimen dried for

2 hr., the optimum bonding range shall be reported as 2 hr. or more.

#### SOFTENING POINT

##### Type of Test

17. The test for softening point is intended for use in determining the temperature at which the cement softens sufficiently to provide an ineffective bond. It consists of subjecting type 2 shear adhesion test specimens (Fig. 1) under a definite shearing load for a specified interval to successive increments of temperature until the bond fails. The temperature at which failure occurs is taken as the softening point. Obviously, the test is useful primarily for comparisons between different cements as the adhesion strength may be seriously reduced at temperatures below the softening point.

##### Preparation of Test Specimens

18. Type 2 shear adhesion test specimens (Fig. 1) using the test cement shall be prepared in duplicate as specified in Section 15, except that the drying time shall be that determined as the optimum bonding range and the specimens shall be aged before test at least 24 hr. under the standard conditions of temperature and humidity (Section 5) without applied pressure.

##### Procedure

19. The test specimens shall be hung with one metal end of each attached to a suitable support in an electric oven complying with the requirements prescribed in Section 4 of the Standard Method of Test for Accelerated Aging of Vulcanized Rubber by the Oven Method (A.S.T.M. Designation: D 573).<sup>3</sup> A weight of 1 lb. (Note) shall be attached carefully without shock to the lower end of each specimen. In attaching the weight and suspending the specimens, suitable clamps and shims shall be used to maintain the specimens in a vertical position and to distribute

the load uniformly over the test specimens. The oven temperature shall be maintained at 100 F. for 15 min. and then increased 15 F. each 20 min. until the rubber separates from the metal. The temperature at which the bond fails shall be reported as the softening point of the test cement used in the specimen.

Note.—With certain special adhesives, it may be desirable to employ very much larger loads than the 1 lb. specified. In these cases, the method may be modified accordingly but any such change must be clearly indicated with the reported results.

### COLD FLOW

#### Type of Test

20. The cold flow test is intended to evaluate the tendency for surfaces bonded by means of the test cement to slip or be displaced when subjected to difference in stress for a sustained period. This yielding is considered to be due to cold flow of the adhesive. The test consists of subjecting a bonded specimen to a specified load for a prolonged period and measuring the elapsed time for failure of the bond.

#### Preparation of Specimens

21. Type 2 shear adhesion test specimens (Fig. 1) using the test cement shall be prepared in duplicate as specified in Section 18.

#### Procedure

22. The test specimens shall be hung with one metal end of each attached to a suitable support and a weight of 1 lb. (see Note, Section 19) shall be applied carefully without shock to the lower end of each specimen. In attaching the weight and suspending the specimens, suitable clamps and shims shall be used to maintain the specimens in a vertical position and to distribute the load uniformly over the test specimens. The loaded specimens

shall be maintained at the standard test conditions of temperature and humidity (Section 5) and observed hourly for 24 hr. or until the time of failure which shall be recorded as the bond failure time in the cold flow test. If no failure occurs in 24 hr., the load shall be increased to 1.5 lb. and the specimens observed for an additional 24 hr. as before. If the bond failure time is over 24 hr. with the 1-lb. load and a second period with the heavier weight is required, the report shall indicate a bond failure time of over 24 hr. under 1 lb. and also shall state the added time under 1.5 lb. required for failure.

### VISCOSITY

23. The viscosity of the cement shall be determined in accordance with the Standard Methods of Test for Viscosity and Total Solids Content of Rubber Cements (A.S.T.M. Designation: D 553),<sup>3</sup> using a duplicate specimen for checking.

### STABILITY

#### Type of Test

24. The stability test provides a means for estimating the relative life of cements in usable form prior to application. When stored, even in containers, many cements jell because of slow vulcanization at ordinary temperatures or other physical or chemical changes and become unusable as adhesives. These changes are evidenced by change in viscosity of the cement. The test consists of subjecting a series of specimens of the cement under specified conditions to an elevated temperature and determining the viscosity at intervals until a marked change occurs which time is taken as a measure of the stability of the cement.

#### Procedure

25. Twelve specimens of 250 ml. each of the test cement shall be measured into

glass test tubes approximately  $1\frac{1}{2}$  in. in diameter and  $9\frac{1}{2}$  in. in length so that each tube is approximately two-thirds full. The tubes shall be fitted with small reflux condensers and placed in a constant temperature water bath at 140 F. The viscosity of the test cement shall be determined and recorded at the start of the test as specified in Section 23. After one week in the water bath, two of the tubes shall be removed, cooled to room temperature and measured for viscosity as at the start of the test. If these specimens have jelled or show a marked increase in viscosity, all of the specimens shall be discarded and new ones started which shall be checked in pairs daily. If the original specimens show no appreciable change in viscosity, the heating shall be continued for five additional days with two specimens taken out each day for viscosity determinations until marked change in viscosity occurs or the cement jells. All viscosity results shall be recorded and reported. The number of days of heating to produce the change shall be reported as the stability number of the cement.

NOTE.—With some special cements made from synthetic rubbers, the viscosity may increase progressively and the cement may become unusable before showing any sudden marked change indicating jelling. In such cases, it may be desirable to test a series of specimens daily in the specified manner and plot the viscosity *versus* time. The resulting curve may be used as the report and no stability number stated. The evaluation of the usability of the cement will depend upon the purpose for which it is intended and the materials to be bonded.

#### COLD BRITTLINESS

##### Type of Test

26. The test for cold brittleness is intended for use in evaluating and comparing the resistance of cements to cracking or chipping when bent at low temperatures. A coat of the test cement on an aluminum strip is cooled at

specified temperatures for a definite time after which the strip is bent in a prescribed manner and examined visually for cracking or chipping of the cement coat.

##### Procedure

27. Six strips of aluminum alloy 1 in. in width by 3 in. in length by 0.040 in. in thickness shall be cleaned and cemented on one side forming a film of adhesive 0.001 in. in thickness as specified in Section 10. The film shall completely cover the one side of the metal. After being aged for at least 3 hr. under the standard atmospheric conditions (Section 5), two of the strips shall be placed in cold atmospheres at each of the following temperatures: 0 F., - 20 F., and - 40 F. The strips shall be maintained at these temperatures for 30 min. and then immediately bent double over a mandrel  $\frac{3}{8}$  in. in diameter with the cemented side out and without removing them from the cold atmosphere. The coatings shall then be examined for visual signs of cracking or chipping. A qualitative statement of the results with the cement at each temperature shall be reported.

#### WEIGHT PER GALLON

##### Apparatus

28. The weight per gallon of a cement shall be determined by use of a pycnometer calibrated for a volume of 83.3 ml. at  $77 \pm 5$  F.

##### Procedure

29. The pycnometer shall be weighed in grams accurately to three significant figures. It shall then be filled with the calibration volume of the test cement which shall have been stirred until homogeneous and shall be free from air bubbles and the filled weight in grams shall be recorded. The weight per gallon of the cement shall be calculated by

dividing the difference in weight of the filled and empty pycnometer by 10.

#### PLASTIC DEFORMATION

##### Type of Test

30. The test for plastic deformation is applicable only to heavy adhesives such as doughs, sealers, putties, etc. It is not suitable nor of use in the case of liquid adhesives.

##### Procedure

31. Two small spheres of the heavy adhesive approximately 1 in. in diameter shall be prepared by rolling in the hands. These spheres shall be placed on small watch glasses and heated in an air oven at 250 F. for 1 hr. The spheres shall

then be removed and examined for flow or distortion. If the spheres have suffered any marked change in shape, the adhesive shall be reported as subject to plastic flow in the deformation test.

#### REPORT

##### Report

32. The report shall include the following:

- (1) Complete identification of the test sample of cement,
- (2) Date of test and date of manufacture if known,
- (3) All observed and recorded data, and
- (4) Statement of the results obtained in each of the tests required.

# *Tentative Method of Test for*

## RESISTANCE OF ADHESIVE BONDS TO CHEMICAL REAGENTS<sup>1</sup>



A.S.T.M. Designation: D 896 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method is intended for the testing of all types of adhesives for resistance to chemical reagents. It includes provisions for reporting loss in strength in accordance with A.S.T.M. methods of test for strength properties of adhesives (Note).

NOTE.—See the Tentative Method of Test for Tensile Properties of Adhesives (A.S.T.M. Designation: D 897)<sup>3</sup> and the Tentative Method of Test for Peel or Stripping Strength of Adhesives (A.S.T.M. Designation: D 903).<sup>3</sup> Methods of test covering other strength properties are in process of formulation by Committee D-14 on Adhesives.

### Apparatus

2. (a) The apparatus shall consist of containers for test specimens and a cabinet for maintaining a temperature between 25 and 30 C. (77 and 86 F.).

NOTE.—Care shall be exercised in choice of materials with respect to adherend and containers in that they are unaffected by the chemicals and solvents used in the test.

(b) Apparatus for making strength tests is specified in the method for the property to be measured (see Note, Section 1).

### Standard Reagents<sup>4</sup>

3. (a) *Sulfuric Acid (30 per cent).*—Slowly add 199 ml. (366 g.) of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) to 853 ml. of water.

(b) *Sulfuric Acid (3 per cent).*—Slowly add 16.6 ml. (30.6 g.) of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) to 988 ml. of water.

(c) *Sodium Hydroxide Solution (10 per cent).*—Dissolve 111 g. of NaOH in 998 ml. of water.

(d) *Sodium Hydroxide Solution (1 per cent).*—Dissolve 10.1 g. of NaOH in 999 ml. of water.

(e) *Ethyl Alcohol (95 per cent).*—Undenatured ethyl alcohol.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-14 on Adhesives.

<sup>2</sup> Accepted by the Administrative Committee on Standards, September 9, 1946.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

<sup>4</sup> Directions for preparation of reagents are for approximately 1-liter quantities. All percentages are by weight.



(f) *Ethyl Alcohol (50 per cent)*.—Add 598 ml. (482 g.) of 95 per cent undenatured ethyl alcohol to 434 ml. of water.

(g) *Acetone*.

(h) *Ethyl Acetate, c.p.*

(i) *Ethylene Dichloride*.

(j) *Carbon Tetrachloride, c.p.*

(k) *Toluene*.

(l) *Heptane*.—Commercial grade, boiling range 90 to 100 C.

(m) *Sodium Chloride Solution (10 per cent)*.—Add 107 g. of NaCl to 964 ml. of water.

(n) *Distilled Water*.—Freshly prepared.

#### Supplementary Reagents<sup>4</sup>

4. (a) *Nitric Acid (10 per cent)*.—Add 108 ml. (153 g.) of HNO<sub>3</sub> (sp. gr. 1.42) to 901 ml. of water.

(b) *Hydrochloric Acid (10 per cent)*.—Add 239 ml. (283 g.) of HCl (sp. gr. 1.19) to 764 ml. of water.

(c) *Acetic Acid (5 per cent)*.—Add 48 ml. (50.5 g.) of glacial acetic acid (sp. gr. 1.05) to 955 ml. of water.

(d) *Oleic Acid, c.p.*

(e) *Ammonium Hydroxide (10 per cent)*.—Add 375 ml. (336 g.) of NH<sub>4</sub>OH (sp. gr. 0.90) to 622 ml. of water.

(f) *Sodium Carbonate Solution (2 per cent)*.—Add 55 g. of Na<sub>2</sub>CO<sub>3</sub>·10H<sub>2</sub>O to 964 ml. of water.

(g) *Hydrogen Peroxide Solution (3 per cent, or U.S.P. 10 volume)*.—Add 98 ml. (108 g.) of commercial grade (100 volume or 28 per cent) hydrogen peroxide to 901 ml. of water.

NOTE.—Additional reagents may be substituted for or supplemented to those listed in Section 4 on agreement between the purchaser and the manufacturer, provided such reagents are within the general scope of this method.

#### Test Specimens

5. The test specimens shall be identical with those required in A.S.T.M. methods of test for the strength properties to be measured (see Note, Section 1).

#### Procedure

6. (a) Each specimen shall be placed in a separate container and totally immersed in a sufficient quantity of the reagent for 7 days at a temperature between 25 and 30 C. (77 and 86 F.) (Note). The specimen shall be placed on edge in the container in the case of flat specimens so that it is supported at an angle from the bottom and side wall of the container. The reagent shall be stirred every 24 hr. by moderate manual rotation of the container.

NOTE.—Choice of an alternate test temperature is permissible upon agreement between the purchaser and the manufacturer.

(b) The individual specimen shall be removed from the reagent, the aqueous solution rinsed off with distilled water and wiped with a clean dry cloth. The strength of the specimen shall then be determined immediately at a temperature between 25 and 30 C. (77 and 86 F.) in accordance with the specified A.S.T.M. method (see Note, Section 1).

#### Report

7. The report shall include the following information for each adhesive tested in all the standard reagents and any specified supplementary reagents:

- (1) Strength property of each specimen,
- (2) Percentage change in average strength during immersion for 7 days calculated to the nearest 0.01 per cent, taking the average strength property of untreated test specimens as 100 per cent.
- (3) General appearance and behaviour of each specimen during and after immersion,
- (4) Type of specimen,
- (5) Trade name and type of adhesive used,
- (6) A.S.T.M. designation of materials and test procedure used, and
- (7) Application, drying, and curing conditions used in preparing specimens

*Tentative Recommended Practice for*  
**DETERMINING THE EFFECT OF ARTIFICIAL  
(CARBON-ARC TYPE) AND NATURAL LIGHT  
ON THE PERMANENCE OF ADHESIVES<sup>1</sup>**



A.S.T.M. Designation: D 904 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Scope**

1. (a) This recommended practice is intended to define conditions for the exposure of adhesives in the form of glued transparent or translucent assemblies to (1) artificial and (2) natural light sources. Where such information is of value, the same exposure conditions may be used on adhesive film or any other suitable form in which light may be a deteriorating factor.

(b) This recommended practice is limited to the method of obtaining the exposure conditions and procedure to be followed, but does not cover methods of test to be used in evaluating the effects of the exposure.

**Apparatus**

2. The apparatus shall consist of the following:

(a) *Artificial Light Exposure Unit.*—

The exposure unit<sup>3</sup> shall consist of a carbon-arc light source and suitable specimen supports, with means being provided for measuring and controlling the current-voltage of the light source, temperature of the air surrounding the specimens, and exposure cycle.

(1) *Light Source.*—A carbon-arc with suitable transparent globe or window.

(2) *Specimen Supports.*—Supports shall be provided for mounting the specimens vertically and in such a manner that uniform distribution of light is obtained at the specimen. The specimens shall be rotated around the arc to insure further uniform distribution of light. If the specimens are mounted both above and below the horizontal center line of the light source, they may be mounted at an angle with the vertical not greater than 30 deg. so that the light from the arc has a normal

<sup>1</sup> Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee D-14 on Adhesives.

<sup>2</sup> Accepted by the Administrative Committee on Standards, December 31, 1946.

<sup>3</sup> Units which successfully fulfill all requirements of this method are the Atlas Single and Twin Arc Apparatus (Alternating or Direct Current), Atlas Fade Ometer, and the National Carbon Apparatus.

incidence upon the specimens. Specimens above and below the horizontal line shall be transposed periodically to provide uniform distribution of the light over the face of the specimens.

(3) *Thermometer*.—A suitable shielded thermometer for determining the temperature of the air at the position of the specimen in the drum.

(b) *Natural Sunlight Exposure Rack*.—The rack shall consist of any suitable framework on which the test specimens may be fastened at an angle of 45 deg. facing south. The specimens shall be protected from direct contact with other weathering elements and foreign matter by means of a transparent shield<sup>4</sup> which will allow transmission of normal solar radiation, but no attempt shall be made to control temperature or relative humidity surrounding the specimens. The roof of a building is a satisfactory location for an exposure rack.

### Test Specimens

3. Test specimens shall be prepared and aged prior to test according to the recommendations of the manufacturer for use of the adhesive. These specimens shall be of a suitable form and number to meet the requirements of the investigation and shall conform in detail with the requirements prescribed in the Tentative Method of Test for Tensile Properties of Adhesives (A.S.T.M. Designation: D 897),<sup>5</sup> the Tentative Method of Test for Peel or Stripping Strength of Adhesives (A.S.T.M. Designation: D 903),<sup>5</sup> or any other A.S.T.M. method of test pertaining to strength properties of adhesives for the desired strength test or with other established test methods such as for light transmission, haze, etc. Where visual inspection or arbitrary

evaluation is resorted to, the specimens may be of any designated shape and size or in the form of a film.

### Method of Test

4. (a) *Artificial Light*.—The test specimens shall be firmly fastened in their holders and then exposed to the carbon-arc light source. Check specimens, shielded from the light source, shall also be provided to determine any degradation resulting from temperature and humidity effects rather than radiation alone. The temperature of the air surrounding the specimens shall be maintained between 35 and 50 C. as measured by the shielded thermometer. Recommended exposure time shall be 10 hr. or a multiple of 10 hr. unless a significant change can be expected in a shorter time. Exposure period may be repeated as often as desired. The units shall at all times be operated under the following conditions:

(1) *Atlas Single and Twin Arc Apparatus (Alternating Current)*.—The average for each trim or burning period shall be 135 v.  $\pm$  2 per cent and 16 amp.  $\pm$  2 per cent at the arc. During the burning period, the voltage may vary between 125 and 145 v. and the amperage between 15 and 18 amp.

(2) *Atlas Single and Twin Arc Apparatus (Direct Current)*.—The average for each trim or burning period shall be 135 v.  $\pm$  2 per cent and 12 amp.  $\pm$  2 per cent at the arc. During the burning period, the voltage may vary between 130 and 145 v. and the amperage between 11 and 13 amp.

(3) *Atlas Fade Ometer*.—Same as prescribed in items (1) and (2) above.

(4) *National Apparatus*.—The average for each trim or burning period shall be 50 v.  $\pm$  2 per cent and 60 amp.  $\pm$  2 per cent at the arc.

Filters (globes and windows) shall be

<sup>4</sup> Pyrex glass, 9200 PX, has been found satisfactory for the shield.

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

replaced after 2000 hr. of use, or when pronounced discoloration or milkiness develops, whichever occurs first (Note). Filters shall be cleaned each day by washing with detergent and water.

NOTE.—The use of the Tentative Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (A.S.T.M. Designation: D 749),<sup>5</sup> is suggested for checking the uniformity of operation of the light source.

(b) *Natural Light*.—The specimens shall be attached to the 45 deg. angle rack and the transparent shield placed in position. Check specimens shielded from the sun shall also be provided to determine any degradation resulting from temperature and humidity effects rather than radiation alone. Specimens shall be examined at 2-week intervals and final evaluation or testing conducted at a predetermined time. A suitable exposure period is one year, although it may be shorter or longer as agreed upon by the manufacturer and the purchaser. The transparent shield shall be cleaned and inspected at least once

every two weeks and replaced at the first signs of discoloration or milkiness.

#### Report

5. The report shall include the following:

(1) Complete identification of the adhesives and specimen materials used,

(2) Method of preparation of specimens, including thickness of glue line or film,

(3) Type and duration of exposure to light including a complete description of the exposure unit used for artificial light, and the geographical location, dates of the exposure period, and general climatic conditions for natural light.

(4) Description of any visual changes in appearance that may have occurred during each exposure period for both the test and check specimens,

(5) Results of any physical, chemical or other tests made to determine the extent of degradation resulting from the exposure. This shall also include tests on the check specimens. The test methods used shall be adequately described.

# *Tentative Method of Test for*

## PEEL OR STRIPPING STRENGTH OF ADHESIVES<sup>1</sup>



A.S.T.M. Designation: D 903 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### INTRODUCTION

The accuracy of the results of strength tests of adhesive bonds will depend on the conditions under which the bonding process is carried out. Unless otherwise agreed upon by the manufacturer and the purchaser, the bonding conditions shall be prescribed by the manufacturer of the adhesive. In order to insure that complete information is available to the individual conducting the tests, the manufacturer of the adhesive shall furnish numerical values and other specific information for each of the following variables:

(1) Procedure for preparation of surfaces prior to application of the adhesive, including the moisture content of wood, the cleaning and drying of metal surfaces, and special surface treatments such as sanding which are not specifically limited by the pertinent test method.

(2) Complete mixing directions for the adhesive.

(3) Conditions for application of the adhesive including the rate of spread or thickness of film, number of coats to be applied, whether to be applied to one or both surfaces, and the conditions of drying where more than one coat is required.

(4) Assembly conditions before application of pressure, including the room temperature, length of time, and whether open or closed assembly is to be used.

(5) Curing conditions, including the amount of pressure to be applied, the length of time under pressure and the temperature of the assembly when under pressure. It should be stated whether this temperature is that of the glue line, or of the atmosphere at which the assembly is to be maintained.

(6) Conditioning procedure before testing, unless a standard procedure is specified, including the length of time, temperature, and relative humidity.

A range may be prescribed for any variable by the manufacturer of the adhesive if it can be assumed by the test operator that any arbitrarily chosen value within such a range or any combination of such values for several variables will be acceptable to both the manufacturer and the purchaser of the adhesive.

### Scope

#### 1. This method of test is intended for

determining the comparative peel or stripping characteristics of adhesives when tested on standard sized specimens and under defined conditions of pre-treatment, temperature, and testing machine speed.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-14 on Adhesives.

<sup>2</sup> Accepted by the Administrative Committee on Standards, December 3, 1946.

### Description of Terms

2. (a) *Peel or Stripping Strength*.—The average load per unit width of bond line required to separate progressively one member from the other over the adhered surfaces at a separation angle of approximately 180 deg. and at a separation rate of 6 in. per min. It is expressed in pounds per inch of width.

(b) *Flexible*.—The designation "flexible" in this test indicates a material of the proper flexural strength and thickness to permit a turn back at an approximate 180-deg. angle in the expected loading range of the test without failure. In order to fulfill all terms of the definition, at least one of the adhered materials must be flexible.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Testing Machine*.—A power-driven machine, preferably of the inclination balance or pendulum type, which shall fulfill the following requirements:

(1) The applied tension as measured and recorded shall be accurate within plus or minus 1 per cent.

(2) Specimens shall be held in the testing machine by grips which clamp firmly and prevent slipping at all times.

(3) The rate of travel of the power-actuated grip shall be 12 in. per min. This rate which provides a separation of 6 in. per min. shall be uniform throughout the tests.

(4) The machine shall be operated without any device for maintaining maximum load indication. In pendulum-type machines, the weight lever shall swing as a free pendulum without engagement of pawls.

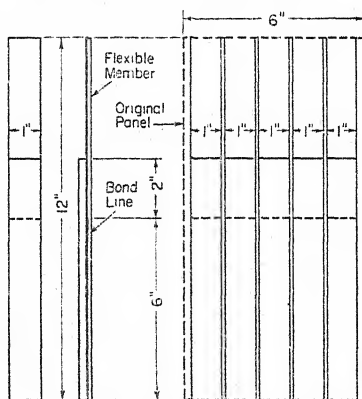
(5) The machine shall be autographic giving a chart having the inches of separation as one axis and applied tension as the other axis of coordinates.

(6) The machine shall be of such

capacity that the maximum applied tension during test shall not exceed 85 per cent nor be less than 15 per cent of the rated capacity.

(b) *Conditioning Room or Desiccators*.—A conditioning room capable of maintaining a relative humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F., or desiccators filled with a saturated salt solution (Note) to give a relative humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F. are required for the conditioning of some specimens.

NOTE.—A saturated salt solution of calcium nitrate will give approximately 51 per cent relative humidity at the testing temperature.



(a) Test Specimen. (b) Specimens from Bonded Panel.

FIG. 1.—Test Specimen.

### Test Specimen

4. (a) The test specimen, shown in Fig. 1(a), shall consist of one piece of flexible material, 1 by 12 in., bonded for 6 in. at one end to one piece of flexible or rigid material, 1 by 8 in., with the unbonded portions of each member being face to face.

(b) In order to maintain a separation rate of 6 in. per min. the specimen shall be relatively nonextensible in the expected loading range. Where a material is sufficiently extensible to lessen radically the separation rate, it shall be backed up with a suitable nonextensible

material. In reporting such a test, the backing material and method shall be completely identified.

(c) Test materials shall be thick enough to withstand the expected tensile pull but not over  $\frac{1}{8}$  in. in thickness. Wherever possible, the standard thickness of specimens shall be: metals,  $\frac{1}{16}$  in., plastics,  $\frac{1}{16}$  in., woods,  $\frac{1}{8}$  in., rubber compounds, 0.075 in., and cotton duck, 30 oz. per sq. yd. Other special materials, as well as the standard materials, shall be completely identified in the test report as specified in Section 9.

(d) At least ten test specimens shall be tested for each adhesive.

(e) Any specimen whose test result is out of line due to some obvious flaw shall be discarded and retest made.

#### Preparation of Test Specimen

5. (a) Any preconditioning or special preparation of the areas to be bonded shall be done in accordance with the recommendations of the manufacturer of the adhesive.

(b) All bonding shall be done in accordance with the procedure and recommendations as outlined by the manufacturer of the adhesive.

(c) While individual specimens may be prepared, it is recommended that specimens be cut from bonded panels approximately 6 in. in width as shown in Fig. 1(b), so that five standard 1-in. wide specimens may be obtained from each panel.

#### Conditioning

6. (a) All specimens shall be conditioned for seven days by exposure to a relative humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F. or until equilibrium is reached, except where the adhesive manufacturer may specify such an aging period to be unnecessary or a shorter period to be adequate.

(b) Special conditioning procedures

may be used by agreement between the purchaser and the manufacturer.

#### Procedure

7. (a) Testing shall be conducted as soon as possible after removal of the test specimens from the conditioning atmosphere and preferably under the same conditions.

(b) The free end of the 1-in. wide flexible member shall be separated by hand from the other member for a distance of about 1 in. The specimen shall then be placed in the testing machine by clamping the free end of the 8-in. long member in one grip, turning back the free end of the flexible member and clamping it in the other grip as shown in Fig. 2. The separated end of the

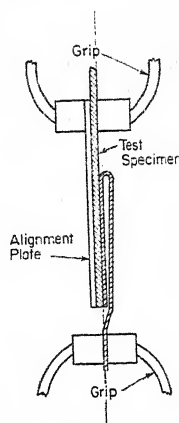


FIG. 2.—Specimen Under Test.

specimen, with all separate parts except the one under test securely gripped, shall be attached to the recording head by means of a clamp using care to adjust it symmetrically in order that the tension shall be distributed uniformly. Provision shall be made to maintain the specimen during test approximately in the plane of the clamps. This may be done either by attaching the minimum weight required to the free end of the specimen or by holding the specimen against an alignment plate (Fig. 2)



attached to the stationary clamp. In either case the added weight shall be taken into account in determining the load causing separation. The 1-in. wide flexible member shall be gripped symmetrically and firmly without twisting in the power-actuated clamp. The autographic mechanism and chart shall be adjusted to zero and the machine started. The separating member shall be stripped from the specimen approximately at an angle of 180 deg. and the separation continued for a sufficient distance to indicate the peel or stripping value. At least one half of the bonded area shall be peeled, even though a peel or stripping value may be indicated before this point.

#### Calculations

8. (a) The actual peel or stripping strength shall be determined by drawing on the autographic chart the best average load line which will accommodate the recorded curve. The load so indicated, corrected for any tare weight which may have been used with the specimen as described in Section 7 (b), expressed in pounds per inch of width for separation at 6 in. per min. shall be reported as the peel or stripping strength for the particular specimen under test.

(b) For each series of tests, the

arithmetic mean of all the values obtained shall be calculated and reported as the "average value."

#### Report

9. The report shall include the following:

(1) Complete identification of the adhesive and specimen tested, including types, source, manufacturer's code numbers, form, etc.,

(2) Method of preparing test specimens,

(3) Conditioning procedure used,

(4) Testing room conditions,

(5) Number of specimens tested,

(6) Speed of testing,

(7) Average value of peel or stripping strength,

(8) Maximum and minimum strength values of the series,

(9) Individual test values, individual autographic charts, and other statistical data requested by the purchaser, and

(10) Type of failure; whether in adhesion, cohesion in the adhesive or in the material being bonded (Note).

NOTE.—Cohesive or adhesive failure may be determined by observation. A cohesive failure is one which has occurred in the adhesive or specimen material itself. Adhesive failure refers to the lack of adherence to the materials being bonded.

# *Tentative Method of Test for*

## TENSILE PROPERTIES OF ADHESIVES<sup>1</sup>



A.S.T.M. Designation: D 897 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### INTRODUCTION

The accuracy of the results of strength tests of adhesive bonds will depend on the conditions under which the bonding process is carried out. Unless otherwise agreed upon by the manufacturer and the purchaser, the bonding conditions shall be prescribed by the manufacturer of the adhesive. In order to insure that complete information is available to the individual conducting the tests, the manufacturer of the adhesive shall furnish numerical values and other specific information for each of the following variables:

(1) Procedure for preparation of surfaces prior to application of the adhesive, including the moisture content of wood, the cleaning and drying of metal surfaces, and special surface treatments such as sanding which are not specifically limited by the pertinent test method.

(2) Complete mixing directions for the adhesive.<sup>3</sup>

(3) Conditions for application of the adhesive including the rate of spread or thickness of film, number of coats to be applied, whether to be applied to one or both surfaces, and the conditions of drying where more than one coat is required.

(4) Assembly conditions before application of pressure, including the room temperature, length of time, and whether open or closed assembly is to be used.

(5) Curing conditions, including the amount of pressure to be applied, the length of time under pressure and the temperature of the assembly when under pressure. It should be stated whether this temperature is that of the glue line, or of the atmosphere at which the assembly is to be maintained.

(6) Conditioning procedure before testing, unless a standard procedure is specified, including the length of time, temperature, and relative humidity.

A range may be prescribed for any variable by the manufacturer of the adhesive if it can be assumed by the test operator that any arbitrarily chosen value within such a range or any combination of such values for several variables will be acceptable to both the manufacturer and the purchaser of the adhesive.

### Scope

1. This method of test is intended for

determining the comparative tensile properties of adhesives when tested on standard shape specimens and under defined conditions of pretreatment, temperature, and testing machine speed.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-14 on Adhesives.

<sup>2</sup> Accepted by the Administrative Committee on Standards, September 9, 1946; introduction added November 23, 1946.

### Description

2. Tensile strength is the maximum tensile load per unit area of original cross-section carried by a test specimen of the types herein described during a tension test. It is expressed in pounds per square inch.

### Apparatus

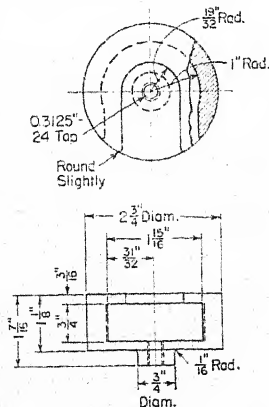
3. The apparatus shall consist of the following:

(a) *Testing Machine*.—A testing machine capable of maintaining a specified rate of loading and comprising essentially the following:

(1) *Fixed Member*.—A fixed or essentially stationary member, carrying one grip.

(2) *Movable Member*.—A movable member, carrying a second grip.

(3) *Grips*.—Grips for holding a test specimen between the fixed member and the movable member. These shall be of the self-aligning type. That is, they shall be attached to the fixed and movable member, respectively, in such a way that they will move into alignment as soon as any load is applied, so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly. While the design of grips of this type is optional one that has been found satisfactory is shown in Fig. 1.



Corrosion Preventive Finish

FIG. 1.—Test Grips (Cold-Rolled Steel).

### (b) Conditioning Room or Desiccators.

—A conditioning room capable of maintaining a relative humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F., or desiccators filled with a saturated salt solution (Note) to give a relative humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F.

NOTE.—A saturated salt solution of calcium nitrate will give approximately 51 per cent relative humidity at the test temperature.

### Test Specimens

4. (a) Test specimens for wood-to-wood adhesives shall conform to the form and dimensions shown in Fig. 2 (a). Hard maple (*acer saccharum* or

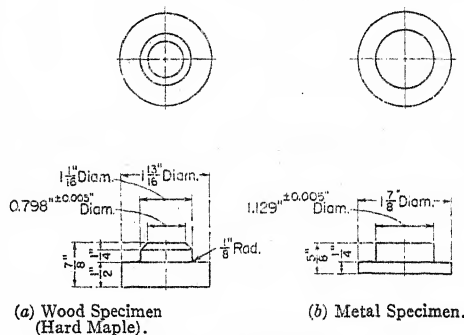


FIG. 2.—Test Specimens.

NOTE.—Surfaces of metal specimens shall be ground flat and parallel. It is preferable to grind all metal specimens at the same time.

*acer nigrum*) having a minimum specific gravity of 0.65 based on oven-dry weight and volume, shall be used. This wood shall be of straight grain and free from defects including small knots, birdseye, short grain, and unusual discolorations within the test area. Blocks  $2\frac{1}{2}$  in. in width, 11 in. in length, and  $\frac{7}{8}$  in. in thickness, from which test specimens are to be made, shall be surfaced immediately before gluing, and may be conditioned prior to surfacing as recommended by the manufacturer. If no moisture conditioning process is recommended by the manufacturer, blocks at the time of surfacing shall be at equilibrium condition (Section 6 (a)) resulting from exposure to a relative

humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F. The blocks shall be surfaced by the standard procedure as follows: Blocks shall be weighed and assembled in pairs so that blocks of approximately the same specific gravity are glued together. The blocks shall be planed smooth, preferably with a hand-feed jointer, and the surfaced faces laid together. The surfaces shall be planed just before gluing, left unsanded, and shall be free from dirt. Blocks shall then be glued as described in Section 5 and conditioned to their original moisture content, after which test specimens conforming to Fig. 2 (a) shall be prepared. For machining to the dimensions specified in Fig. 2 (a) pieces 2 by 2 in. shall be sawed from the glued hard maple block.

(b) Test specimens for metal-to-metal adhesives shall conform to the form and dimensions shown in Fig. 2 (b). Metal specimens may be reused after testing by resurfacing by grinding flat and parallel those areas which contain the adhesive. Metals conforming to the following specifications of the American Society for Testing Materials are recommended:

Metal	A.S.T.M. Designation <sup>a</sup>
Brass .....	B 16
Copper .....	B 133, type A
Aluminum .....	B 211, alloy CG21
Steel .....	A 108, grade 1020
Phosphor bronze .....	B 139, grade B2
Magnesium .....	B 107, alloy AZ61X or M1
Nickel Silver .....	B 151, alloy B

<sup>a</sup> These designations refer to the following specifications:

Specifications for Free-Cutting Brass Rod and Bar for Use in Screw Machines (A.S.T.M. Designation: B 16).<sup>1</sup>  
Specifications for Copper Rods, Bars, and Shapes (Tentative) (A.S.T.M. Designation: B 133).<sup>2</sup>  
Specifications for Aluminum and Aluminum-Alloy Bars, Rods, and Wire (Tentative) (A.S.T.M. Designation: B 211).<sup>3</sup>  
Specifications for Cold-Finished Carbon-Steel Bars and Shafting (A.S.T.M. Designation: A 108).<sup>4</sup>  
Specifications for Phosphor Bronze Rods, Bars, and Shapes (Tentative) (A.S.T.M. Designation: B 139).<sup>2</sup>  
Specifications for Magnesium-Base Alloy Bars, Rods, and Shapes (Tentative) (A.S.T.M. Designation: B 107).<sup>5</sup> and  
Specifications for Copper-Nickel-Zinc Alloy Rod, Bar, and Wire (Tentative) (A.S.T.M. Designation: B 151).<sup>3</sup>

<sup>1</sup> 1946 Book of A.S.T.M. Standards, Part I-B.

<sup>4</sup> 1946 Book of A.S.T.M. Standards, Part I-A.

(c) At least 10 and preferably 20 specimens shall be tested for each adhesive in the case of wood-to-wood adhesives. At least 10 test specimens shall be tested for each adhesive in the case of metal-to-metal adhesives.

(d) Specimens that break at some obviously fortuitous flaw remote from the glue line shall be discarded and retest made, unless such flaws constitute a variable the effect of which it is desired to study.

### Gluing

5. (a) Gluing shall be done in accordance with the procedure as outlined by the manufacturer of the adhesive.

(b) For wood specimens a pair of maple blocks, selected as described in Section 4 (a),  $2\frac{1}{2}$  in. in width, 11 in. in length, and  $\frac{7}{8}$  in. in thickness shall be glued together with the grain of the wood parallel in each piece.

(c) For metal specimens preparation of areas which are to be cemented shall be in accordance with the recommendations of the manufacturer of the adhesive.

### Conditioning

6. (a) Unless otherwise agreed upon by the purchaser and the manufacturer, all wood specimens shall be preconditioned by exposure to a relative humidity of  $50 \pm 5$  per cent at  $77 \pm 2$  F. until they reach equilibrium, as determined by no progressive change in weight.

(b) Preconditioning is not required for metal-to-metal bonds. The adhesive is ready for test when it has been applied in accordance with Section 5.

(c) Special conditioning procedures may be used by agreement between the purchaser and the manufacturer.

### Procedure

7. (a) It is preferable that the testing be carried out under the same conditions as prescribed in Section 6.

(b) The specimen shall be placed in the grips of the testing machine, care being taken to align the specimen and the grips with an imaginary line joining the points of attachment of the grips to the machine. Wood-to-wood specimens shall be inserted so that the grain direction is at right angles to the long horizontal axis of the slots in the grip. Loading of the specimen should be started as quickly as possible.

(c) *Speed of Testing.*—Tension tests for all materials shall be made by applying a load to the specimen of 600 to 700 lb. per min., or the crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. (1.27 mm. per min.) when the machine is running idle.

(d) *Record.*—Record the maximum load carried by the specimen at failure and also the following:

(1) In the case of wood-to-wood adhesives record the percentage of glue failures, wood failures, and contact failures. This will be based on a visual inspection.

(2) In the case of metal-to-metal adhesives record the percentage of cohesion, adhesion, and contact failures (Note). This will be based on a visual inspection.

NOTE.—Cohesive failure may be obtained by observing how much of the failure has occurred in the adhesive itself. That is, if the cement has adhered to the metal test pieces and no voids are visual, it represents a 100 per cent cohesive failure. Adhesion failure refers to the lack of

adhering to metals being fastened. Contact failure refers to lack of glue lines being in contact due to uneven surfaces, poor pressure distribution, etc.

### Calculations

8. (a) Tensile strength of the wood-to-wood specimens shall be calculated by multiplying the breaking load by two. This result shall be expressed in pounds per square inch and if possible reported to three significant figures.

(b) Tensile strength of the metal-to-metal specimens shall be the breaking load and shall be expressed in pounds per square inch and if possible reported to three significant figures.

(c) For each series of tests the arithmetic mean of all the values obtained shall be calculated and reported as the 'average value.'

### Report

9. The report shall include the following:

(1) Complete identification of the material tested, including types, source, manufacturer's code numbers, form, etc.,

(2) Method of preparing test specimens,

(3) Conditioning procedure used,

(4) Testing room conditions,

(5) Number of specimens tested,

(6) Speed of testing, and

(7) Average value of the tensile strength, with an average value of the 'percentage of glue failure for the wood specimens or cohesion failure for the metal specimens.

# Tentative Method of Test for

## ABSORPTION BY BIBULOUS PAPERS OF WATER AND WRITING INK<sup>1</sup>



A.S.T.M. Designation: D 824 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method<sup>3</sup> of test covers the procedure for determining the rate at which unsized and absorbent papers will absorb water or ink. It is especially useful in testing the absorbency of toweling or blotting papers. It is not intended for use on sized papers of any kind (Note). This method is based on the determination of the length of time required to absorb completely a definite quantity of water or ink.

NOTE.—The absorption test of sized papers should be made in accordance with the Standard Method of Test for Time of Penetration by Water of Sized Paper and Paper Products (Dry Indicator Method) (A.S.T.M. Designation: D 779).<sup>4</sup>

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> For further information on this method the following references may be consulted:

E. O. Reed, "Determining the Absorbency of Paper," *Paper*, Vol. 21, No. 19, p. 14 (January 16, 1918).

F. T. Carson and F. V. Worthington, "New Types of Equipment for Testing Paper," *Paper Trade Journal*, Vol. 95, No. 16, p. 59 (October 20, 1932).

F. T. Carson, "Testing Paper for Permeability to Liquid," *Paper Trade Journal*, Vol. 80, No. 10, p. 59 (March 5, 1925).

B. W. Scribner, "Standards for Paper Towels," *Circular No. 407*, Nat. Bureau of Standards (1935).

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

### Apparatus

2. The apparatus shall consist of the following:

- (a) *Pipette*.—A 1-ml. measuring pipette<sup>5</sup> graduated in divisions of 0.01 ml.
- (b) *Screen*.—A 4-mesh wire screen.
- (c) *Support*.—A suitable support.
- (d) *Watch Glass*.
- (e) *Stop Watch*.

### Test Liquids

3. (a) *Water*.—For testing water absorption, distilled water is preferable, but water of ordinary purity (tap water) may be used.

(b) *Standard Ink*.—For ink absorption, use a standard ink having the following composition:

Tannic acid, g. . . . .	11.7
Gallic acid crystals, g. . . . .	3.8
Ferrous sulfate, g. . . . .	15.0
Hydrochloric acid dilute, g. . . . .	12.5
Carbolic acid (phenol), g. . . . .	1.0
Soluble blue (Schultz No. 539, Color Index 707), g. . . . .	3.5
Water. . . . .	to make a volume of 1000 ml. at 20 C. (68 F.)

<sup>5</sup> S. W. Reese and M. A. Voutz, "A Water Absorbency Tester Using 0.1 cc. of Water," *Paper Trade Journal*, Vol. 100, No. 7, p. 33 (February 14, 1935).

All chemicals used in preparing the ink shall be of c.p. quality. Some blue dyes react with carbolic acid to cause a film having a metallic appearance and such dyes shall not be used. The ink shall be prepared as follows: Dissolve the tannic acid and gallic acid in about 400 ml. of water at about 50 C. (122 F.). In a separate vessel, dissolve the ferrous sulfate in about 200 ml. of water which contains the hydrochloric acid. In a third vessel, dissolve the dye in about 200 ml. of water. Mix the three solutions in a 1-liter volumetric flask, rinse the vessels with small portions of water, and add the rinsings to the flask. Add the carbolic acid. Dilute with water to 1 liter at 20 C. (68 F.).

#### Test Specimen

4. Each test specimen shall consist of a single sheet of paper approximately 4 by 4 in. The test specimens shall be cut from the sample in such a way as to be thoroughly representative of it.

#### Procedure

5. (a) The test shall be made in the atmospheric conditions and on conditioned paper in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>4</sup>

(b) Place the test specimen on the 4-mesh wire screen, which is slightly dished and supported by the edges, so that the liquid remains in a pool in the center. Fill the 1-ml. measuring pipette with the test liquid at a temperature of  $23 \pm 2$  C. ( $73 \pm 3.5$  F.). Hold the pipette at an angle of about 30 deg. with

the horizontal with the tip nearly in contact with the paper and allow the specified amount of the test liquid to flow on the specimen. For blotting paper, exactly 1 ml. of the standard ink shall be used and the time of delivery shall not be less than 4 nor more than 6 sec. For other bibulous papers, such as paper toweling, 0.1 ml. of water shall be employed. While the liquid is flowing, keep the tip of the pipette in the drop until delivery is completed.

(c) For samples having a slow rate of absorption, that is, absorption time over 120 sec., cover the test specimen with a watch glass to prevent evaporation.

(d) Measure with a stop watch the time of absorption in seconds, from the start of flow of liquid until it is completely absorbed as indicated by no further reflection of light from it when viewed at an angle. An equal number of tests shall be made on each side of the paper and not less than ten specimens shall be tested.

#### Report

6. The report shall include the following:

- (1) The nature of the test liquid employed,
- (2) The amount of the test liquid completely absorbed, and
- (3) The minimum, maximum, and average absorption time in seconds.

#### Reproducibility

7. The reproducibility of average test results secured by this method is usually within 5 per cent.



## *Tentative Method of Test for* ADHESIVENESS OF GUMMED TAPE<sup>1</sup>



A.S.T.M. Designation: D 773 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers the procedure for determining the adhesiveness of gummed sealing tape not less than 2 in. in width, used for sealing shipping containers, when tested under the conditions specified herein.

NOTE.—The test results are indicative of how well the tape will adhere and whether it will adhere with satisfactory rapidity in the sealing operation. The results do not necessarily indicate how a given tape will behave when stuck to other surfaces than the Kraft paper specified, nor how well it will maintain its adhesiveness over a period of time.

### Apparatus

2. The testing apparatus shall be the Harnden-McLaurin gummed tape tester which consists of the following:

(a) Two adjacent platens in the same

horizontal plane forming a table-like top divided in the center and balanced, and so pivoted that, on releasing them, the adjacent edges tilt upward and apart.

(b) A means for actuating the platens.

(c) A means of holding the test paper firmly in place on top of the platens with a tilting clamp across each platen, the front edge of each clamp being 2 in. from and parallel to the division between the platens.

(d) A scale with pointer for registering the retardation of the swing of the platens which results from the resistance of the gum of the tape.

(e) A roll-feed, lever-operated moistener for moistening the gum. The moistener shall have a single brush set in a pan of water so constructed as to maintain a constant water level. The depth of the water shall be not less than 1 in., and the brush shall extend 1.7 in. (43 mm.) above the surface of the water.

(f) A motor-driven roller with automatic timing for pressing the test specimen on the test paper. It shall be made of iron, 3½ in. in width, 4½ in. in diameter, having a covering of sponge rubber ¼ in. in thickness on the circumferential surface, and shall weigh 16½ lb.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

For further information on this method the following references should be consulted:

George H. Harnden, "Determining the Adhesive Strength of Gummed Tape," ASTM BULLETIN, Am. Soc. Testing Mats., No. 98, May, 1939, p. 23.

C. G. Weber, "Testing the Adhesive Strength of Gummed Paper Sealing Tape," Modern Packaging, Vol. 15, No. 4, December, 1941, p. 66.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

(g) A device for feeding a test specimen which shall be set to deliver a piece of moistened tape  $5.5 \pm 0.1$  in. in length.

(h) A mechanism for synchronized automatic operation of the moving parts.

### Calibration of Apparatus

3. Each new testing machine shall have been calibrated by the manufacturer against the master calibrating instrument at each 10-point interval in the range of 15 to 85, and shall be recalibrated by the manufacturer whenever found necessary.

### Test Paper

4. (a) The test paper<sup>3</sup> shall be gummed Kraft conforming to the following requirements:

	Maximum	Minimum
Basis weight (24 by 36 in., 480 sheets), lb. ....	95	85
Thickness, in. ....	0.008	0.0074
Bursting strength, points. ....	...	90
Smoothness (Bekk tester), each side, sec. ....	35	15
Water resistance (dry-indicator method), sec. ....	175	125

(b) The sheets of test paper shall be 6 by 9 in. with the grain parallel to the longer dimension.

(c) The sheets of test paper shall be conditioned separately for not less than 12 hr. according to the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>4</sup>

### Test Specimens

5. (a) The test specimens shall consist of not less than 24 strips, each  $5\frac{1}{2}$  in. in length, and cut during the test from each sample roll of tape to be tested.

(b) The sample rolls of gummed tape shall not be conditioned for testing and

shall be kept tightly wound until the test is begun.

### Procedure

6. (a) The test shall be made in an atmosphere maintained in accordance with A.S.T.M. Method D 685.

(b) The apparatus shall be examined to make certain that the machine is level in both directions, that the pointer gives a zero reading when the machine is operated without load, and that the moistening device contains distilled water at the same temperature as the testing room.

(c) The moistening brush shall be prepared for testing by washing in lukewarm water, and then soaking it for 30 min. in distilled water. If the brush is in continuous use, it shall be cleaned after testing each sample roll of tape, and at least every 2 hr.

(d) A sheet of the test paper shall be placed on the table of the machine, clamped securely, and with a sharp knife slit through the center over the opening between the platens. The test specimen shall then be cut from the sample roll, discarding 15 layers before cutting the specimen. The roll shall be placed in the moistener and a strip of tape  $5\frac{1}{2}$  in. in length fed over the moistening brush by operating the feed lever. The test specimen shall be cut immediately and laid lightly on the test paper, taking care to center it lengthwise over the cut in the test paper. The machine automatically passes the roll over the test specimen and trips the platens. The interval between the cutting of the test specimen and the tripping of the mechanism shall be 5 sec. with one cycle of rolling. At least 24 consecutive specimens of each sample roll shall be tested, making an equal number of tests on each side of the test paper.

(e) Scale readings below 15 units or above 85 units are not regarded as dependable. Results shall be disregarded

<sup>3</sup> The testing paper may be obtained from the Gummed Industries Assn., 19 W. 44th Street, New York, N. Y.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

if the test specimen breaks or the pendulum hesitates.

**Report**

7. The report shall include the following:

(1) The average, minimum, and maximum points of adhesive strength, expressed to the nearest whole number, and

(2) Width of the tape tested in inches.

## *Tentative Method of Test for*

### EDGE TEARING STRENGTH OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 827 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. This method of test covers the procedure for determining the edge tearing strength of paper. A strip of paper of a specified size is simultaneously torn at opposite points at the edges of the strip by means of a thin V-notched beam held in a stirrup which is fastened in one of the clamps of a tension testing machine. The use of this test shall be limited to papers which will fold evenly over the V-notched beam.

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Edge Tear Stirrup*.—A Finch edge tear stirrup (Fig. 1) shall be used, attached to a suitable pendulum type tension testing machine. The edge tear stirrup consists of a thin steel plate which forms a horizontal beam supported on edge by the ends of a stirrup-shaped

frame. The thin metal tang of the stirrup frame is fastened in the lower clamp of the tension testing machine so that the vertical center line of the stirrup coincides with the line connecting the mid-points of the upper and lower clamps. The horizontal beam is removable from the stirrup frame and two beams of different thicknesses are furnished for use with papers of different thickness ranges. The edge of the beam forms a shallow V-notch, the sides of which subtend an angle  $150 \pm 1$  deg. The sides of the V-notch are semicircular in cross-section and are formed either by filing and scraping with a semicircular-shaped scraper or by soldering a drill rod of a diameter equal to the thickness of the beam, to the edges of the V-notch. In either case the edges of the V-notch shall be smooth and straight.

(b) *Tension Testing Machine*.—The tension testing machine shall be of the pendulum type, power or hand driven, preferably equipped with two or three different pendulum weights and cor-

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

responding scales of different capacities, including scales with capacities of 0 to 1 kg. (0 to 2.2 lb.), and 0 to 5 kg. (0 to 11 lb.). It shall conform to the requirements prescribed in Section 2 of the Tentative Method of Test for Tensile Breaking Strength of Paper and Paper Products (A.S.T.M. Designation: D 828).<sup>3</sup> It shall be calibrated as described in Section 3 of A.S.T.M. Method D 828.

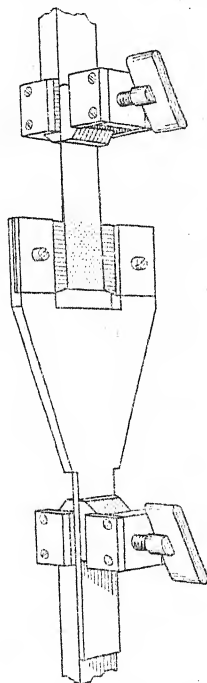


FIG. 1.—Edge Tear Stirrup.

### Test Specimens

3. (a) The test specimens shall be cut from samples selected as prescribed in the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585).<sup>3</sup> If desired and if possible, the specimens shall be cut in the two principal directions of the sheet (Note). Specimens shall be strips cut straight and parallel with smooth edges and shall

be not more than 2.54 cm. (1.00 in.) nor less than 1.5 cm. (0.59 in.) in width and not less than 25 cm. (10 in.) in length, preferably somewhat longer.

NOTE.—Since the specimen is stressed in a very limited area the results are subject to a wider variation than in a test such as the tensile strength test. For this reason, it is desirable to test at least ten specimens cut from either or both principal directions of the sheet.

(b) The test specimens shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>3</sup>

### Procedure

4. (a) Make the test in an atmosphere conditioned in accordance with A.S.T.M. Method D 685.

(b) *Beam Thickness*.—Attach a beam of the proper thickness to the stirrup frame. The beam thickness shall be selected with reference to the paper thickness as follows:

Paper Thickness, in.	Beam Thickness, in.
0.030 in. and under.....	0.050 $\pm$ 0.002
Over 0.030.....	0.100 $\pm$ 0.002

(c) Fasten the thin tang of the stirrup in the lower clamp (Note) of the tension testing machine so that the vertical center line of the stirrup coincides with the line connecting the mid-points of the upper and lower clamps of the testing machine, and so that the sides of the V-notch are symmetrically located with the line through the mid-points of the clamps.

NOTE.—The stirrup may be fastened in the upper clamp, if desired. This procedure will require rebalancing the tension testing machine to compensate for the weight of the stirrup.

(d) Place the lower clamp of the machine so that the lower edge of the upper clamp is about 9 cm. (3.5 in.) above the V-notched beam.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(e) Thread the test specimen through the stirrup, under the beam, and bring the two ends together and fasten them in the upper clamp. In this operation, most of the slack in the specimen is taken up, but care shall be exercised not to apply sufficient stress to start tearing the specimen.

(f) Apply a tearing force to the specimen by applying the load at a rate conforming to that prescribed in Section 5(c) of A.S.T.M. Method D 828, namely, that the rate of loading shall be adjusted so that tearing occurs in not less than 5 nor more than 15 sec. Make the application of the first increments of load to the specimen very slowly, if possible,

to minimize abnormal strains due to inertia effects.

### Report

5. The report shall include the following:

(1) The average, minimum, and maximum edge tearing strengths in kilograms or pounds (to two significant figures) for each of the two principal directions of the sheet tested,

(2) The thickness of the beam used,

(3) Rate of travel of lower clamp,

(4) The width and thickness of the test specimens, and

(5) The number of specimens tested.

# Tentative Method of

## QUALITATIVE EXAMINATION OF MINERAL FILLER AND MINERAL COATING OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 686 - 46 T

ISSUED, 1942; Revised, 1943, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers the procedure for the qualitative determination of the mineral constituents of filled and coated papers.

### Reagents

2. (a) *Hydrochloric Acid* (sp. gr. 1.12).—Mix 500 ml. of reagent grade HCl (sp. gr. 1.19) with 400 ml. of distilled water. (A 30 per cent solution of H<sub>2</sub>O<sub>2</sub> may be used in proportionately lesser quantity.)

(b) *Sulfuric Acid* (sp. gr. 1.84).—Reagent grade H<sub>2</sub>SO<sub>4</sub>.

(c) *Diluted Sulfuric Acid* (5:95).—Into 950 ml. of distilled water, pour 50 ml. of H<sub>2</sub>SO<sub>4</sub> (sp. gr. 1.84) while stirring constantly.

(d) *Acetic Acid* (sp. gr. 1.058).—Reagent grade glacial HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>.

(e) *Ammonium Hydroxide* (sp. gr. 0.90).—Reagent grade NH<sub>4</sub>OH.

(f) *Sodium Hydroxide Solution*.—Dissolve 100 g. of c.p. NaOH in 1 liter of distilled water.

(g) *Ammonium Sulfate*.—C.p. (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> crystals.

(h) *Ammonium Carbonate Solution*.—Dissolve 250 g. of c.p. (NH<sub>4</sub>)<sub>2</sub>CO<sub>3</sub>·H<sub>2</sub>O in 1 liter of distilled water and add 100 ml. of NH<sub>4</sub>OH (sp. gr. 0.90).

(i) *Ammonium Chloride Solution*.—Dissolve 100 g. of c.p. NH<sub>4</sub>Cl in 1 liter of distilled water.

(j) *Barium Chloride Solution*.—Dissolve 100 g. of BaCl<sub>2</sub>·2H<sub>2</sub>O in 1 liter of distilled water.

(k) *Cobalt Nitrate Solution*.—Dissolve 10 g. of c.p. Co(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O in 100 ml. of distilled water.

(l) *Hydrogen Peroxide* (3 per cent).—Prepare a solution of U.S.P. H<sub>2</sub>O<sub>2</sub> containing about 3 per cent H<sub>2</sub>O<sub>2</sub> by weight.

(m) *Potassium Ferrocyanide Solution*.—Dissolve 15 g. of c.p. K<sub>4</sub>Fe(CN)<sub>6</sub>·3H<sub>2</sub>O in 1 liter of distilled water.

(n) *Potassium Dichromate Solution*.—Dissolve 49 g. of c.p. K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> in 1 liter of distilled water.

(o) *Magnesium Reagent*.—Dissolve 0.5 g. of para-nitrobenzeneazorescorcinol in NaOH (1 per cent) and dilute to 100 ml. with ethyl alcohol (95 per cent).

(p) *Sodium Carbonate* (Na<sub>2</sub>CO<sub>3</sub>).—c.p., powdered.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Latest revision accepted by the Society at annual meeting, June, 1946.



(q) *Microcosmic Salt Solution*.—Dissolve 50 g. of  $\text{NaNH}_4\text{HPO}_4$  in water and dilute to 1 liter.

(r) *Lead Acetate Paper*.—Prepare lead acetate indicator paper by immersing strips of filter paper in a fresh saturated solution of c.p.  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ .

(s) *Platinum Wire*.

(t) *Charcoal Block*.

### Test Specimens

3. The test specimens shall be cut from the sample in such a way as to be thoroughly representative of the paper. Each specimen shall be of sufficient size to yield at least 0.1 g. of ash.

### Procedure

4. (a) If separate analyses of filling materials and coating materials are desired, remove the coating from the test specimens by the procedure described in the Standard Method for Quantitative Determination of Coating on Mineral-Coated Paper (A.S.T.M. Designation: D 687) of the American Society for Testing Materials.<sup>3</sup> Evaporate to dryness the aqueous mixture thus obtained and make analyses on this residue and on the base stock.

(b) Tests for components that may alter their form on ashing shall be made on unignited test specimens as follows:

(1) To a portion of the unignited coating or specimen, add  $\text{HCl}$  (sp. gr. 1.12) with caution; effervescence indicates the presence of a carbonate, which may be checked in the usual way by testing the evolved gas with lime water.

(2) To a small portion of the  $\text{HCl}$  solution, add a drop or two of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution; a green coloration indicates sulfite.

(3) Heat the contents of the beaker to boiling and test the fumes with

moistened lead acetate paper; the development of a metallic gray or black color indicates the presence of sulfide.

NOTE.—So far as is known, mixtures of sulfides and sulfites are not used in loading or coating paper. To test for  $\text{CO}_2$  in the presence of sulfites, add to the contents of the beaker a weak solution of iodine (about 0.1 N) until the liquid becomes yellow. Sulfites are oxidized to sulfates. Test the evolved gas for  $\text{CO}_2$  in the usual manner with lime water.

(c) Ignite a test specimen in accordance with the procedure described in the Standard Method of Test for Ash Content of Paper and Paper Products (A.S.T.M. Designation: D 586) of the American Society for Testing Materials.<sup>3</sup> Divide the ash into three portions, each of which shall contain not less than 0.05 g. of mineral matter, and place two portions in separate 250-ml. beakers.

(d) To one portion of the ash (Paragraph (c)), add 10 g. of  $(\text{NH}_4)_2\text{SO}_4$  and 20 ml. of  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84). Cover with a watch glass, boil vigorously for at least 3 min., allow to cool, and examine as follows:

(1) Considerable undissolved matter indicates the presence of aluminum and/or magnesium silicates, and/or diatomaceous earth. If the solution is clear, aluminum or magnesium silicates and diatomaceous earth are absent. (Calcium and barium sulfates will not be in an undissolved state unless the sample being tested is more than 0.5 g.)

(2) Add water cautiously to the cool solution and adjust the volume to about 125 ml.; the formation of a precipitate indicates the presence of barium sulfate.

(3) Add  $\text{H}_2\text{O}_2$  (3 per cent) to the solution, which will turn a yellow to orange color if titanium is present, the color developed being dependent on the amount of titanium present.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(4) If there is a residue when the sample is more than 0.5 g. or a precipitate from the treatment of the ash with  $\text{H}_2\text{SO}_4$  (Paragraph (d)), filter, wash with  $\text{H}_2\text{SO}_4$  (5:95), and make a flame test with the platinum wire; a green flame indicates the presence of barium, a red flame indicates calcium, and a yellow to colorless flame indicates aluminum or magnesium silicates, or both.

(e) To the second portion of the ash (Paragraph (c)), cautiously add  $\text{HCl}$  (sp. gr. 1.12); an effervescence indicates the presence of a carbonate or sulfite. (This is a check on the addition of the acid to a portion of the original paper.)

(1) To a small portion of this solution, add a drop or two of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution; a green coloration indicates sulfite.

(2) Heat the contents of the beaker to boiling and test the fumes with moistened lead acetate paper; the development of a metallic gray or black color indicates the presence of sulfites. (This test checks that made on the original specimen of paper. Sulfates may be reduced to sulfides, or sulfides oxidized to sulfates, depending on ignition conditions.)

(f) If there is an insoluble residue from the treatment of the ash with  $\text{HCl}$  (Paragraph (e)), decant the solution through a filter paper and dilute the filtrate to a volume of 125 to 150 ml. Divide the resulting solution into four portions.

(1) To the first portion add  $\text{BaCl}_2$  solution; a precipitate indicates the presence of sulfates soluble in  $\text{HCl}$ .

(2) To the second portion add  $\text{K}_4\text{Fe}(\text{CN})_6$  solution; the presence of a white precipitate indicates zinc.

(3) To the third portion add one or two drops of the magnesium reagent and make alkaline with  $\text{NaOH}$  solution; magnesium forms a sky-blue

precipitate while all other minerals commonly used in paper give a violet coloration to the solution. Take care not to add in excess of the reagent, since it is liable to mask the blue precipitate. In this case, filter the solution and examine the filter paper for the presence of a blue precipitate.

(4) To the fourth portion add an excess of  $\text{NH}_4\text{OH}$  and  $\text{NH}_4\text{Cl}$  solutions. Aluminum will appear as a white floc if it is present in acid-soluble form. Filter and add  $(\text{NH}_4)_2\text{CO}_3$  solution to the filtrate. Barium and calcium will be indicated by a white precipitate. Without filtering, make the solution acid with acetic acid (which dissolves the precipitate), after which add  $\text{K}_2\text{Cr}_2\text{O}_7$  solution. If barium is in acid-soluble form, its existence will be shown by the formation of a yellow precipitate. Filter and add an excess of  $(\text{NH}_4)_2\text{CO}_3$  solution. Since barium is removed by the preceding step, a white precipitate indicates calcium. This may be checked by a flame test.

(g) Mix the third portion with an excess of sodium carbonate on a charcoal block, and moisten with a small amount of cobalt nitrate solution. Aluminum will give a permanent blue coloration upon heating with a blowtorch flame. (Care should be taken not mask the blue coloration by the use of an excess of cobalt nitrate solution.)

(h) Place the unfolded filter paper containing the acid-insoluble residue (Paragraph (f)) in a platinum crucible (Note 1). Dry and ignite with free access of air until all organic matter is removed. Add 1 to 2 g. of  $\text{Na}_2\text{CO}_3$  and fuse until a clear melt is obtained or all reaction has ceased. Decompose the melt in 25 ml. of hot water, heat to boiling, and wash thoroughly with water. Reserve the filtrate and first two washings for analysis of the water soluble

portion according to (1) below. Wash the insoluble portion from the filter paper back into the original beaker with a stream of water, place the beaker under the funnel and pour 10 ml. of hot reagent grade HCl (sp. gr. 1.18) through the filter paper. Reserve the contents of the beaker for analysis of the water insoluble portion according to (2) below.

(1) Acidify the water soluble portion with HCl, evaporate to dryness, and bake at 100 to 105 C. for 1 hr. Moisten the residue with reagent grade HCl (sp. gr. 1.18), let stand for a few minutes, add 5 to 10 ml. of diluted HCl and warm. The presence of silicate is indicated by a light, flocculent insoluble residue in the solution, best observed against a dark background. Filter and add  $\text{BaCl}_2$  solution to a portion of the filtrate. A precipitate indicates sulfates. To another portion of the filtrate add  $\text{NH}_4\text{OH}$ . A precipitate indicates aluminum.

(2) Warm the acid solution of the water-insoluble portion, add 50 ml. of water, boil and add a very slight excess of  $\text{NH}_4\text{OH}$  to precipitate aluminum (and iron). Filter and wash, reserving the precipitate for analysis according to (3) below. Add 5 ml. of dilute  $\text{H}_2\text{SO}_4$  (5:95). A precipitate indicates barium. Boil and filter. Add a slight excess of  $\text{NH}_4\text{OH}$  to the filtrate followed by ammonium oxalate solution to insure the absence of calcium, filtering if any precipitate results. Cool the solution and add 5 ml. of  $\text{NH}_3\text{OH}$  and an excess of microcosmic salt solution. Stir well. If a precipitate forms within 15 min., the presence of magnesium is indicated.

(3) Wash the insoluble precipitate obtained by the  $\text{NH}_4\text{OH}$  treatment in (2) above with water and transfer it to the original beaker with a stream of water (Note 2). Add 5 ml. of HCl and warm. If solution is not complete, add 3 ml. of reagent grade  $\text{H}_2\text{SO}_4$  (sp. gr. 1.84) and heat under a hood until  $\text{SO}_3$  fumes are

formed. Cool and dilute to 35 ml. Any barium sulfate not decomposed by the  $\text{Na}_2\text{CO}_3$  fusion will be insoluble and should be removed by filtration. Make solution neutral with NaOH solution plus an additional volume equal to that of the neutralized solution. Boil, cool and filter. An insoluble precipitate indicates titanium. Neutralize the filtrate with HCl (sp. gr. 1.12), boil and add a slight excess of  $\text{NH}_4\text{OH}$ . A precipitate indicates aluminum.

NOTE 1.—The procedure described in Paragraph (h) will serve as a check on the examination as outlined in Paragraphs (a) to (g).

NOTE 2.—The procedure described in (3) is necessary only if both titanium and HCl-insoluble aluminum compounds are present, and it is desired to estimate the relative amounts of each.

### Interpretation of Results

5. (a) Carbonates together with considerable acid soluble calcium indicate the presence of calcium carbonate which may exist as chalk or whiting. If HCl-soluble magnesium is also present, a mixture of calcium and magnesium carbonates is indicated. A combination of barium and carbonate may exist as witherite. Barium in this form will be shown in the HCl-treated portion of the ash, as barium sulfate is insoluble in diluted HCl (sp. gr. 1.12).

(b) The use of sulfides as filling or coating materials in the paper industry is restricted to zinc sulfide alone or in combination with barium sulfate (lithopone). A positive test for zinc in the absence of sulfide indicates the use of zinc oxide in the paper. A combination of the oxide and the sulfide cannot be identified as such.

(c) A positive test for acid-soluble sulfates and calcium indicates the use of calcium sulfate as crown filler, gypsum, or satin white, etc. If considerable HCl-soluble aluminum is also present in the coating, the mineral used may be

satin white. If carbonates are used in conjunction with sulfides, there may be a positive test for sulfates when none are present.

(d) A positive test for calcium and sulfite indicates the presence of calcium sulfite.

(e) The presence of considerable magnesium and silicate indicates the use of talc, agalite, or asbestine. Silica may also indicate the presence of diatomaceous earth. The characteristic diatom forms may be readily recognized on microscopic examination.

(f) The residue from the portion of the ash treated with  $H_2SO_4$  (sp. gr. 1.84) may be clay, talc, diatomaceous earth, or a mixture of these substances. A positive test for aluminum indicates that clay was used.

(g) Barium sulfate (barytes or blanc

fixe) is indicated by the formation of a precipitate on the dilution of the  $H_2SO_4$  solution and by a positive flame test for barium on the residue.

(h) Titanium may be present as titanium dioxide alone or combined with barium or calcium sulfates. If domestic clays are used, a faint test for titanium may be obtained as titanium may be present in small amounts in such clays.

### Report

6. Report all cations and anions and radicles found present indicating the relative amounts of each, such as trace, slight amount, considerable, large amount, etc. The report should also interpret the results of the analysis in terms of the fillers or mineral coating materials indicated to be present.

### EXPLANATORY NOTE

NOTE.—The following references may be of interest in connection with this method of analysis:

R. C. Griffin, "Technical Methods of Analysis," Second Edition, McGraw-Hill Book Co., Inc., New York, N. Y. (1923).

L. J. Curtman, "Qualitative Chemical Analysis," Macmillan Co., New York, N. Y. (1936).

F. P. Treadwell and W. T. Hall, "Analytical Chemistry," Ninth Edition, John Wiley & Sons, Inc., New York, N. Y. (1937).

J. P. Mehlig and K. R. Johnson, "Test for Magnesium with *p*-Nitrobenzeneazoresorcinol," *Industrial and Engineering Chemistry, Analytical Edition*, Vol. 12, January 15, 1940, p. 30.

# *Tentative Methods of Test for*

## PLY ADHESION OF PAPER OR VULCANIZED FIBRE<sup>1</sup>



A.S.T.M. Designation: D 825 - 46 T

ISSUED, 1945; REVISED, 1946.<sup>2</sup>

These Tentative Methods have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. These methods of test cover procedures for measuring the amount of bond or adhesion between the plies of a multiply paper, such as that made on a cylinder type of paper machine, or of vulcanized fibre up to and including 0.032 in. in thickness.

### METHOD A. FOR PAPER

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Testing Machine.*—The essential features of a satisfactory type of testing machine (Fig. 1) are as follows: The base of the testing machine, *A*, shall consist of a horizontal metal frame about 2 ft. in length and 6 in. in width, inside of which two jaw carriages, *B* and *C*, may be made to move horizontally in ball bearings. The two jaws, *D* and *E*, 3 in. in width set in the carriages 6 in. in length are such that they may approach and touch each

other. By means of a crank, *F*, attached by a reversed direction cord, *G*, to the left jaw carriage, *B*, the left jaw carriage may be slowly pulled away from the right jaw carriage. The right jaw carriage, *C*, is attached by a cord, *H*, to the periphery of a 6-in. diameter grooved face wheel, *J*. Pendulum weight arms, *K*, of definite mass and length are attached to the axle of this wheel. The axle turns in ball bearings. The circumference of the wheel bears a graduated scale ranging from 0 to 110 in 5-unit divisions. The frame of the testing machine is equipped with leveling glasses for the two principal directions and leveling screws at each end. Zero reading is indicated by a stationary pointer when the tester frame is level, the weight arms are freely hanging vertically, and the right jaw carriage is far to the right. As this carriage is moved to the left and the cord tension turns the wheel clockwise, an increasing force is indicated until, when the weight arms are about horizontal,<sup>2a</sup> the maximum

<sup>1</sup> Under the standardization procedure of the Society, these methods are under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

<sup>2a</sup> Editorially corrected in January, 1947. The position of the weight arms formerly was incorrectly given as "vertical."

scale reading of 110 is indicated. Each unit on the scale is equivalent to 7.62 g. of force, this ratio being such that when testing a specimen 3 in. in width, adhesive force per centimeter width of the specimen is indicated in grams.

(b) *Cutting Board*.—A paper-cutting board large enough for cutting strips 3 in. in width and 8 in. in length.

#### Calibration of Testing Machine

3. The accuracy of the graduated scale on the testing machine may be determined by disconnecting the cord

#### Test Specimens

4. Samples shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685),<sup>3</sup> after which at least 5 test specimens shall be cut 3 in. in width by approximately 8 in. in length, with the length perpendicular to the machine direction of the paper.

#### Procedure

5. Ply separation across the 3-in. width of a specimen shall be started by

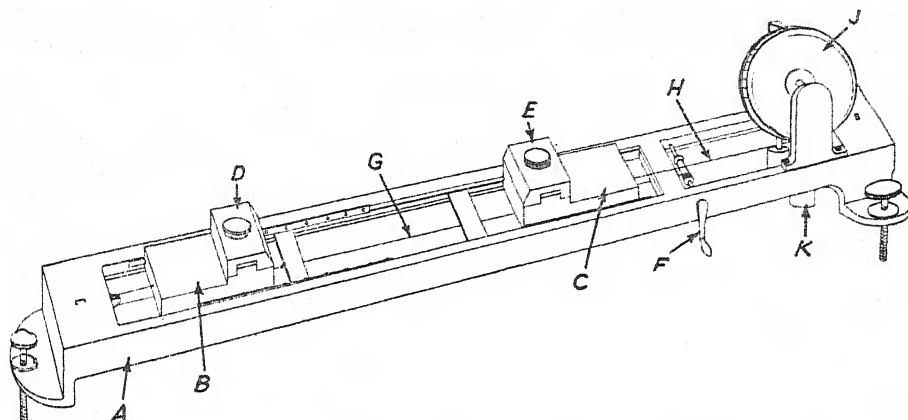


FIG. 1.—Ply Adhesion Testing Machine, Method A.

from the right jaw carriage and by passing it clockwise around the circumference of the wheel and vertically downward tangentially over the right-hand side of the wheel. A hole in the frame of the testing machine and in the table supporting the testing machine permits this cord to be loaded with various standard weights. The observed scale readings shall be compared with the correct readings calculated as follows:

$$R = \frac{W}{7.62}$$

where:

$R$  = correct scale reading, and  
 $W$  = weight applied in grams.

hand as near as possible to the center lamination of the paper. A sharp-pointed lead pencil may be used for this purpose. After the delamination has been carried across the 3-in. width, it shall be continued for about  $\frac{3}{4}$  in. of the 8-in. length of the specimen. These separated ends shall be folded back against the outside of the test specimen for convenience in inserting them in the jaws of the testing machine. The two jaws of the testing machine shall be brought together with the wheel indicator in the zero position. One of the separated sections shall be clamped in each of the jaws, the unseparated portion

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

remaining in an upright position at right angles to the jaw surfaces. (The specimen is then shaped like an inverted T.) The vertical unseparated section of the paper shall be allowed to lie between the forefinger and the second finger of the left hand in order to maintain it perpendicular to the separated parts during the entire test. The crank shall be turned with the right hand at the rate of one revolution per second until the jaws begin to separate, at which point the indicated adhesion shall be noted. In order to check this result, the weight arms shall be returned to a vertical position by reversing the direction of rotation of the crank and the delamination started again and continued for a notation of the uniformity and the amount of ply adhesion (in grams per centimeter width as indicated on the scale). In case of excessively high ply adhesion, that is, values greater than 110 g. per cm. width, a specimen 1.5 in. in width may be tested, in which case the indicated scale readings shall be multiplied by 2.

### Report

6. The report shall include the following:

(1) Maximum, minimum, and average ply adhesion in grams per centimeter of width, and

(2) The width of specimen tested.

### METHOD B. FOR VULCANIZED FIBRE

#### Apparatus

7. The apparatus shall consist of the following:

(a) *Tension Testing Machine*.—The tension testing machine shall be of the pendulum type, power or hand driven, preferably equipped with two or three different pendulum weights and corresponding scales of different capacities,

including scales with capacities of 0 to 1 kg. (0 to 2.2 lb.), and 0 to 5 kg. (0 to 11 lb.). The jaws shall be 2 in. in width and not more than 5 in. apart. It shall conform to the requirements prescribed in Section 2 of the Tentative Method of Test for Tensile Breaking Strength of Paper and Paper Products (A.S.T.M. Designation: D 828).<sup>3</sup> It shall be calibrated as described in Section 3 of A.S.T.M. Method D 828.

(b) *Cutting Board*.—A paper-cutting board large enough to cut specimens of vulcanized fibre 2 in. in width and at least 8 in. in length.

(c) *Straight-Edge*.

(d) *Knife*.—A sharp-pointed knife.

#### Test Specimens

8. (a) At least two specimens shall be tested. The test specimens shall be cut 2 in. in width and at least 8 in. in length, the length being in the machine direction of the vulcanized fibre.

(b) About 1 in. from one end of each test specimen, a straight cut shall be made across one face of the specimen, parallel to the end and of a uniform depth of approximately half the thickness. The short end of the specimen shall be grasped between the thumb and the bent forefinger of the left hand and the specimen shall be bent back over the forefinger, so as to open the cut. The vulcanized fibre along the cut edge opposite the short end of the specimen shall be picked apart with the knife blade so as to separate the plies at the middle, until the separated edge can be grasped between the thumb and forefinger of the right hand. With the separated ends held horizontally between the thumbs and bent forefingers of the two hands, and the long, unseparated portion of the specimen held vertically between the remaining bent fingers of the hands, the specimen shall be further separated into



two halves, using the middle fingers as a fulcrum, until the separation is even all the way across the specimen between the middle plies and has progressed for at least 3 in. By varying the angle between the separated portions and the unseparated part of the specimen, the depth of tear can be regulated. If the separation is uneven, the pulling shall be manipulated until it is even. This can be verified by holding the specimen up to the light, when the diffusion of light through the separated portions should be uniform. When proper separation has been accomplished, the unseparated 1-in. piece shall be cut off.

(c) The specimens, after initial separation as specified above, shall be conditioned for at least 24 hr. as prescribed in the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>3</sup>

#### Procedure

9. With the separated ends of the test specimen held in the jaws of the testing machine, the unseparated portion of

the specimen shall be held out at right angles by hand and the machine started. To maintain the 90-deg. relationship between the separated and unseparated portions of the test specimen during the pulling, it is necessary to hold the unseparated portion lightly between the fingers near the point of separation. The pendulum shall be allowed to swing freely, without use of the pawls. The test specimen shall be separated by the machine for at least 3 in. of its length, and maximum and minimum readings shall be noted. The speed of the movable jaw of the testing machine shall be 12 in. per min.

#### Report

10. The report shall include the following:

(1) Average thickness of the specimens,

(2) Maximum, minimum, and average ply adhesion for each specimen in pounds per 2 in. of width, and

(3) Grade and color of the vulcanized fibre.

## *Tentative Method of Test for* PUNCTURE AND STIFFNESS OF PAPERBOARD, CORRUGATED AND SOLID FIBERBOARD<sup>1</sup>



A.S.T.M. Designation: D 781 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers the procedures for puncture and stiffness tests of paperboard, corrugated, and solid fiberboard.

### PUNCTURE TEST

#### Nature of Test

2. The puncture test is a measure of the energy required to force a puncture head of designated size and shape completely through a sample of fiberboard. The measured value is dependent upon the resistance to puncture, and the stiffness, of the board.

#### Apparatus

3. The apparatus,<sup>3</sup> illustrated in Fig. 1, shall consist of the following:

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

For additional information on this method the following references should be consulted:

R. L. Beach, *Technical Association Papers*, Technical Assn. Pulp and Paper Industry, Series XXII, No. 1, p. 240 (1939).

S. D. Wells, *Paper Trade Journal*, Vol. 116, No. 13, pp. 29-38 (1943).

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> This apparatus is obtainable from the General Electric Co., Special Products Division, Industrial Division, Schenectady, N. Y.

(a) *Pendulum*.—A pendulum incorporating an arm in the form of an arc of 90 deg.

(b) *Puncture Point*.—A puncture point affixed to the end of the rod and having the shape of a right angle triangular pyramid 1 in. in height.

(c) *Pointer Collar*.—A loose collar around the base of the puncture point to eliminate friction resulting from the punctured material rubbing against the puncture arm.

(d) *Trigger*.—A trigger, or latch, to hold the pendulum in the horizontal position.

(e) *Two Clamping Plates*.—Two clamping plates for holding the specimen to be tested.

(f) *Pointer and Scale*.—A pointer and scale to indicate the arc through which the pendulum travels after penetrating through the specimen.

(g) *Weights*.—Weights, which when attached, give a maximum scale value of at least 350 in.-lb. of energy when the pendulum is in the horizontal position, and scale graduations corresponding to the weights used.

(h) *Loose Plate*.—A loose plate which is inserted between the clamping plates when testing material for the purpose of defining the test area. This plate shall be removed when checking the zero reading of the apparatus.

(i) *Adjustable Screw*.—An adjustable screw which bears against the pointer assembly during the measuring half of the pendulum swing, which permits

bearing plate normally inserted between the clamping jaws. *Failure to remove these may result in damage to the instrument.* Set the pointer a few graduations above zero. Raise the pendulum to the cocked position and release it. Note the pointer reading. Repeat this operation several times. The pointer should register zero with each attempt. If such is not the case, make suitable

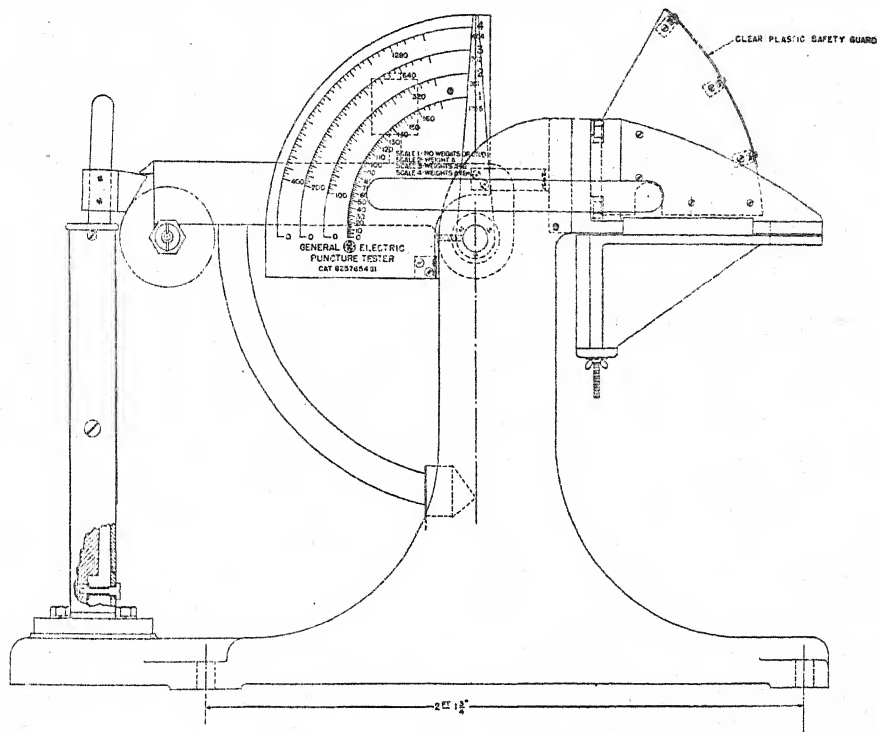


FIG. 1.—Apparatus for Puncture and Stiffness Tests.

adjustment of the zero position of the pointer.

#### Adjustment of Apparatus

4. (a) Place the apparatus on a stable platform and level it in both principal directions. The pendulum should now move substantially free of friction.

(b) To check the zero reading it is important to remove the weights, the weight supporting stud, and the metal

adjustment of the zero adjustment screw (located on the arm extending from the bearing housing of the pendulum) until the zero reading is obtained.

(c) To check the friction of the pointer, set the pointer at zero before releasing the pendulum from the cocked position. After the pendulum has completed its swing, the pointer should not be more than  $\frac{1}{8}$  in. below the zero position. An error of more than  $\frac{1}{8}$

in. indicates excessive friction and can be remedied by oiling the pointer bearing or relieving the spring pressure between the pointer collar and the shaft. This pressure is governed by the position of the screw which is threaded into the projection on the pointer collar.

(d) No point on the scales can be checked except the zero point, and it is not necessary to do so, as all points are measured from the zero point according to the mathematical formula:

$$R = Wg (1 - \cos A)$$

where:

$R$  = energy in inch-pounds required to puncture the material tested,

$W$  = total weight of the pendulum plus any added weights used,

$g$  = distance of the center of gravity of the total pendulum mass from the center of rotation, and

$A$  = angle through which the pendulum swings past the vertical (Note).

NOTE.—The actual numerical values on the scale are not in inch pounds, but have a multiplying factor introduced in order to make the numerical values for the ordinary grades of boxboard more nearly in line with the commonly used values.

(e) The loose sleeve is held in place on the base of the puncture point by a pin containing a small ball under spring pressure. The position of this pin in the puncture point base is adjustable and can be locked by means of a set screw. The position of this pin and ball should be such that the loose sleeve is very lightly held on the base of the puncture point, and can be removed with a minimum of energy.

### Conditioning

5. Test specimens shall be conditioned before testing in accordance with the Standard Method of Conditioning Paperboard, Fiberboard, and Paperboard

Containers for Testing (A.S.T.M. Designation: D 641).<sup>4</sup>

### Procedure

6. (a) Place the test specimen between the clamping plates. Before making each test, place the loose sleeve against the base of the puncture point and set the pointer about 1 in. above the expected reading. Raise the pendulum to the horizontal position. Release by pushing the latch handle to the left. Note the reading on the proper scale after the pendulum has completed its swing. This reading should be made to the nearest one-half division of the scale used. It is not advisable to make tests giving values within 25 per cent of the high end of the scale. In such cases use a higher scale.

(b) Orient the test specimens in such a manner that the edge of the puncture point, which is in the plane of the puncture arm, is perpendicular to the corrugations of combined corrugated board, or grain direction of uncombined sheets or solid fiber board, of half of the specimens, and is parallel to the corrugations, or grain direction, of the other half of the specimens. Make all tests from the outside liner of the corrugated board and from the smooth side when testing uncombined board.

### STIFFNESS TEST

#### Nature of Test

7. The stiffness test is a measure of the energy required to force a puncture head of designated size and shape completely through a sample of fiberboard when the board is prepared to receive the puncture head by slitting in the prescribed manner. The measured value is largely dependent upon the stiffness of the board.

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

## Apparatus

8. The apparatus shall be as described in Section 3 and in addition the following will be required:

(a) *Template*.—A template containing three slits  $2\frac{1}{2}$  in. in length, meeting at one point and so spaced angularly that they define the paths of the edges of the puncture point as it passes through the specimen.

(b) *Sharp Knife*.

## Test Specimens

9. (a) Ten test specimens shall be selected in such a manner that they accurately represent the sample of paper to be tested. The sample shall have been conditioned for testing as described in Section 5.

(b) The specimen shall be of sufficient size that no slippage occurs between it and the clamping jaws of the apparatus. No part of the test area shall be closer than  $1\frac{1}{2}$  in. to any edge, score line, or imperfection in the board.

(c) The template (Section 8(a)) shall be used in locating and defining the cuts in the specimens. Five specimens shall be cut so that the edge of the puncture point which is in the plane of the curved puncture arm is parallel to the corrugations, or grain direction, and five cut with this edge perpendicular to the corrugations, or grain direction. The template shall be laid on the inner liner according to the above orientations and the slits of the template traced with a pencil. The template shall be removed and the cuts defined by the pencil tracing made with a sharp knife. These three lines shall be completely cut through, their ends shall be perpendicular to the surface of the board, and they shall meet completely at the midpoint.

## Procedure

10. (a) Adjust the apparatus as described in Section 4. Place and angularly orient the test specimen between the clamping jaws of the testing machine so that the point and the three edges of the puncture point will pass exactly through the prepared slits.

(b) The weights to be used for each scale are indicated on the scale plate of the apparatus. Select the scale and corresponding weight so that readings will fall as close to the center of the scale as possible.

(c) Make all tests from the outside liner of the corrugated board and from the smooth side when testing uncombined board. Place the loose sleeve around the base of the puncture point before each test is made. (It is designed to prevent the punctured material from bearing against the puncture arm and thus causing an error due to friction.) Set the pointer about 1 in. ahead of the position of the expected reading before each test is performed.

(d) Record only readings in which the puncture head passes completely through the board. Read and record the values to the nearest one-half division of the scale used.

## Report

11. The report for both the puncture and stiffness tests shall include the following:

(1) Room temperature and relative humidity,

(2) The scale used,

(3) Number of tests made each way of board,

(4) The average results for tests made each way of the material,

(5) The total average results, and

(6) The maximum and minimum values obtained.

## *Tentative Method of Test for* RESISTANCE OF PAPER TO PASSAGE OF AIR<sup>1</sup>



A.S.T.M. Designation: D 726-46 T

ISSUED, 1943; REVISED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test is applicable to papers and paper products which permit the passage of 100 cu.cm. of air in 2 to 1800 sec., except those which cannot be clamped securely against surface and edge leakage, such as crepe and corrugated papers.

### Apparatus

2. (a) The apparatus shall consist of an outer cylinder which is partly filled with oil, and an inner cylinder, having an open or closed top, sliding freely in the outer cylinder. Air pressure for the test shall be provided by the weight of the inner cylinder. The apparatus shall be arranged to furnish air pressure to the specimen held between clamping plates having a circular orifice of 1.00 sq. in. in area. The clamping plates may form the top of the inner cylinder, or may be mounted in the base of the apparatus,

the latter construction being preferable. An elastic gasket shall be attached to the clamping plate on the side exposed to air pressure, and the paper specimen shall be held in contact with the gasket when clamped for test. The purpose of the gasket is to prevent leakage of air between the surface of the paper and the clamping plate.

(b) The gasket shall consist of a thin, elastic, oil-resistant, nonoxidizing material, having a smooth surface (Note). The inside diameter of the gasket shall be 1.125 in. and the outside diameter 1.375 in. To align and protect the gasket in use, it shall be cemented with shellac into a groove machined in the clamping plate. This groove shall be concentric with the aperture in the opposite orifice plate. It shall be 1.128 in. in inside diameter and 0.020 in. in depth. Its outside diameter may be 1.385 in. for convenience in inserting and attaching the gasket.

NOTE.—Thiokol, grade ST, polished plate molded,  $\frac{3}{8}$  in. in thickness, 50 to 60 Durometer hardness, is a satisfactory gasket material.

(c) The outer cylinder shall be 10 in. in height, and shall have an internal diameter of 3.25 in. It shall be equipped

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

For further information on this method the following reference may be consulted:

"First Report of the Paper Testing Committee to the Technical Association of the Papermakers' Association of Great Britain and Ireland," 1937, pp. 62-66.

<sup>2</sup> Revision accepted by the Society at annual meeting, June, 1946.

with four bars, each 7.5 in. in length, 0.094 in. in width, and 0.094 in. in thickness, mounted vertically and equidistantly on the inner surface of the outer cylinder to act as guide tracks for the movable cylinder.

(d) The movable inner cylinder shall be graduated in units of 50 cu.cm., and shall have a total range of 350 cu.cm. It shall be 10 in. in height, and shall have an external diameter of 3.00 in. and an internal diameter of 2.916 in. It shall weigh  $567 \pm 1.0$  g.

NOTE.—Some inner cylinders are also graduated in units of 25 cu. cm. for the first 100 cu. cm., and have a graduation at the 400-cu. cm. interval.

(e) The oil used with the apparatus shall be a lubricating oil having a viscosity of 60 to 70 sec. Saybolt Universal at 100 F. (37.8 C.) and a flash point of not less than 275 F. (135 C.).

NOTE.—A light spindle oil is suitable for this purpose. Oil is used in preference to water because it does not affect the moisture content of the sample, nor does it affect the aluminum inner cylinder. The oil should not contain any essential oil, or other easily volatile oil, and for that reason a minimum flash test is specified.

### Calibration of Apparatus

3. The apparatus shall be tested for air leakage by clamping a thin piece of smooth, hard-surface, air-tight material, such as metal foil or cellophane, between the orifice plates, and testing in accordance with the procedure outlined in Section 6. The leakage shall not exceed 50 cu.cm. in 5 hr.

### Sampling

4. The paper to be tested shall be sampled in accordance with Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585).<sup>3</sup>

### Test Specimens

5. (a) Not less than ten test specimens

shall be cut from the sample, each approximately 1.875 in. in width, and 5 in. in length, except that specimens of any size larger than 1.875 in. in both directions may be used in the apparatus having the clamp in the base.

(b) The test specimens shall be conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>3</sup>

### Procedure

6. (a) Place the apparatus on a level surface so that the cylinders are vertical. Fill the outer cylinder with oil to a depth of approximately 5 in. as indicated by a ring marked on the inner surface of the cylinder.

(b) Test not less than ten conditioned specimens in the standard atmosphere prescribed in A.S.T.M. Method D 685, making an equal number of tests (see Section 7, item 1) on each side of the sample.

(c) If using the instrument having the clamp in the top of the inner cylinder, raise the inner cylinder, hold it in a raised position with one hand, clamp the specimen between the clamping plates, then lower the cylinder and allow it to float in the oil.

NOTE.—The proper procedure for clamping the specimen is to tighten the knurled nuts alternately, so that the clamping pressure will be equal on both sides. If only one knurled nut at a time is tightened, the clamp will not bear evenly on the specimen, and air leakage will occur between the surfaces of the clamp and the specimen. If more convenient, the inner cylinder may be removed from the outer cylinder and replaced after the paper has been clamped.

(d) If using the instrument having the clamp in the base, first raise the inner cylinder until its top rim is supported by the catch, clamp the specimen between the clamping plates, then gently lower the inner cylinder until it floats.

(e) When a steady movement of the inner cylinder has been attained, measure

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



with a stopwatch, or other timing device, the number of seconds required for the first two consecutive 50-cu.cm. intervals to pass the rim of the outer cylinder, starting at the 0 mark. With very resistant papers, the reading may be taken at the end of the first 50-cu.cm. interval, and the results doubled. With very open or porous papers, a larger volume of air may be read on the inner cylinder and converted to the 100-cu.cm. standard volume. In cases where it is not possible to obtain a steady movement of the inner cylinder before the 0 mark is reached, the test may be started at the 50-cu.cm. mark.

(f) Take precautions to avoid sub-

jecting the apparatus to vibration, as this increases the rate of air displacement.

#### Report

7. The report shall include the following:

(1) The average number of seconds required for the displacement of 100 cu.cm. of air through an area of 1.0 sq.in., obtained by testing at least five specimens with the felt side up and five specimens with the wire side up.

NOTE.—Smaller or larger volumes of air may be read on the inner cylinder, and converted to the 100-cu. cm. standard volume.

(2) A statement that the gasket, described in Section 2 (b), was used.

# *Tentative Method of Test for* TENSILE BREAKING STRENGTH OF PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 828 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. This method<sup>3</sup> of test covers the procedure for measuring the tensile breaking strength, expressed in units of force of paper and paper products.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> For further information on this method the following references may be of interest:

A. H. Stang and L. R. Sweetman, "Speed Control for Screw-Power Testing Machines Driven by Direct-Current Motors," *ASTM BULLETIN*, No. 87, August, 1937, p. 15.

F. T. Carson, "Some Notes on Revision of Methods for Measuring the Strength of Paper," *Paper Trade Journal*, Vol. 102, No. 19, May 7, 1936, pp. 39-42; *Technical Association Papers*, No. 19, June, 1936, pp. 172-175.

J. d'A. Clark, "Some Observations on Burst, Tensile, and Stretch Tests," *Paper Trade Journal*, Vol. 102, No. 2, January 9, 1936, pp. 40-42; *Technical Association Papers*, No. 19, June, 1936, pp. 264-266 (discussion, pp. 90-94).

R. C. Griffin and R. W. McKinley, "Studies of Tensile and Bursting Tests," *Paper Trade Journal*, Vol. 102, No. 2, January 9, 1936, pp. 34-35; *Technical Association Papers*, No. 19, June, 1936, pp. 222-223.

Institute of Paper Chemistry, "Instrumentation Studies No. 29: Measurement of the Tensile Strength of Paper," February 16, 1940.

C. V. Oliver, "Variability in Results Met With in Paper Strength Testing," *Proceedings, Technical Section, Paper Makers' Association of Great Britain and Ireland*, No. 11, Part I, October, 1930, pp. 53-74 (discussion, pp. 74-84).

Paper Makers' Association of Great Britain and Ireland, Technical Section, Paper Testing Committee, First Report, London, The Association, 1937, 85 pages; *Proceedings, Technical Section*, Vol. 18, Part IA (which contains the discussion), June, 1937; *World's Paper Trade Review*, Technical Convention Number, March, 1937, pp. 4-72.

"Tensile Strength of Paper," (proposed final revision of TAPPI standard T404m), *Paper Trade Journal*, Vol. 102, No. 8, February 20, 1936, p. 133; *Technical Association Papers*, No. 19, June, 1936, p. 271.

## Apparatus

2. The tension testing machine shall have the following features:

(a) Two clamps, the jaw edges of which can be set for distances from  $100 \pm 10$  mm. ( $3.94 \pm 0.39$  in.) to  $180 \pm 10$  mm. ( $7.09 \pm 0.39$  in.) apart. The center lines of the clamps shall be in the same plane parallel to the direction of motion and shall be so aligned that they will hold the test specimen in the same plane throughout the test, without slippage.

(b) Means of applying a gradually increasing load to the test specimen until it breaks, the rate of increase being such that the additional load applied each second is not different from the additional load applied in the previous second by more than 5 per cent. This condition is fulfilled by the usual pendulum-type apparatus, when the lower clamp moves at a constant rate of speed (Note).

NOTE.—To ensure uniform operation an automatic drive is preferable to hand operation. Motor-driven machines of the pendulum, double-scale type, can be adapted to accommodate all but the weak-paper group by means of a two-

step pulley. With the pendulum-type apparatus the rate of loading may be reduced for the weak-paper group either by substituting a lighter weight on the end of the pendulum or else fastening the weight to the pendulum nearer the bearing and recalibrating the scale. If the normal scale is accurate, a single calibration point (taken preferably in the working range) will be sufficient to give the scale conversion factor. A variable speed motor (see footnote 3, first item) is more convenient when many different rates of loading are required. At least two modern tension testing machines are available with variable speed mechanical drives.

(c) Means of indicating the applied load at the instant of specimen-fracture, to an accuracy of plus or minus 1 per cent. To attain this accuracy for the pendulum-type apparatus, the capacity of the apparatus should be selected and adjusted so that the fracture of the specimen will occur: (1) in the pendulum-type apparatus, when the pendulum makes an angle of not less than 4 nor more than 45 deg. with the vertical, or (2) in the spring-actuated type apparatus, when the breaking load indications are in the middle third of the scale.

### Calibration of Apparatus

3. The apparatus shall be accurately leveled in both of its principal directions and the mechanism shall be made to move freely. Apply weights of known value to the upper clamp so that it moves in the same direction and plane as when testing a specimen. In the case of the pendulum-type apparatus, disengage the pawls of the pendulum, suspend the test weight by a flexible connection from the center of the upper clamp, release the latch holding the pendulum at zero indication, and allow the pendulum to come slowly to equilibrium. Note the indicated scale reading for each load applied and record all test data. It is recommended that readings be taken at not less than three distributed points on the scale of the instrument.

### Test Specimens

4. (a) The test specimens shall be representative of the sample obtained as prescribed in the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585)<sup>4</sup> and conditioned as prescribed in the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>4</sup> Prepare a sufficient number of specimens cut from each principal direction of the paper, excluding watermarks, creases, or other visible imperfections.

(b) The specimens shall be clean-cut to within plus or minus 1 per cent of their nominal widths, with edges parallel, and shall be long enough to be inserted in the jaws of the clamps of the testing apparatus without handling the section under test. Specimens shall not exceed 5.08 cm. (2.0 in.) in width and shall preferably be not less than 1.27 cm. (0.50 in.) (Note).

NOTE.—Varying the width of the test specimens between 1.27 and 5.08 cm., with a proportionate change in the rate of loading, does not, in general, make much difference in the proportionate test results except for unbeaten long-fiber papers, when the difference may be appreciable.

### Procedure

5. (a) Make the tests in a conditioned atmosphere as prescribed in A.S.T.M. Method D 685.

(b) Level the testing machine and adjust its zero reading. The ratio of the clearance distance between jaws to the width of the specimens shall be not less than 5 to 1, nor more than 15 to 1.

(c) After placing the test specimen loosely in the jaws of the clamps, align it squarely, then tighten the upper camp, take up slack, and finally tighten the lower clamp, without touching the portion of the specimen under test. Then

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

apply the load as follows: For very weak papers, such as tissues, paper towels, newsprint, and similar papers (in general, papers breaking below 2.3 kg. (5 lb.) per 15 mm. (0.59 in.) width), adjust the loading so that fracture occurs in not less than 5 nor more than 15 sec. For all other papers adjust the rate of loading so that the lower clamp moves at a rate of  $12 \pm 0.5$  in. per min.

(d) Test at least ten specimens cut in each principal direction of the paper.

(e) Reject readings when the specimen slips in the jaws, or fractures in or at the edge of either jaw.

(f) Wherever possible, record each reading to three significant figures.

(g) Correct reading by applying the calibration test results (Section 3).

#### Report

6. The report shall include the following:

(1) Results obtained on specimens cut in the machine direction of the paper

shall be reported as tensile breaking strength, machine direction, and the results obtained on specimens cut in the cross-direction of the paper shall be reported as tensile breaking strength, cross-direction,

(2) Average, minimum, and maximum values, either as kilograms or as pounds, to three significant figures,

(3) Number of specimens tested,

(4) Type and capacity of the apparatus used,

(5) Rate of loading,

(6) Specimen width, and

(7) Distance between jaws of the clamps.

#### Reproducibility

7. Duplicate measurements of the tensile breaking strength of different sets of samples from the same lot of paper, and on different apparatus, should agree within 5 per cent.

## *Tentative Method of Test for* WET TENSILE BREAKING STRENGTH OF PAPER AND PAPER PRODUCTS<sup>1</sup>



A.S.T.M. Designation: D 829 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers a procedure for determining the tensile breaking strength of paper and paper products after wetting with water or other liquid. It is of special usefulness in evaluating the performance characteristics of paper towels, photographic paper, water-resistant wrapping papers, and other papers subjected to stress during processing or use, while wet. Depending on the use requirement, the test specimen may be immersed in the appropriate liquid for a given time and tested whether it is completely saturated or not. However, unless otherwise specified, the wet tensile breaking strength test is assumed to be performed on the test specimen after complete saturation.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Wet Strength Device*.—A Finch wet strength device (Fig. 1) consisting of an inverted stirrup about 3.8 cm. (1.5 in.) in width and about 7.6 cm. (3 in.) in length, made of metal strap by which a horizontal rod, about 2.8 cm. (1.1 in.) in length and  $5 \pm 0.05$  mm. ( $0.188 \pm 0.002$  in.) in diameter, is supported. Between the straps and under the horizontal rod is a small, vertically movable container for holding water or other liquid. The liquid container locks in its uppermost position, so that the horizontal rod is then immersed in the liquid to a depth of at least 1.9 cm. (0.75 in.). A thin metal tang forming the lower part of the inverted stirrup permits it to be fastened in the lower clamp of a tension testing machine.

(b) *Tension Testing Machine*.—The tension testing machine shall be of the pendulum type, power or hand driven, and designed for testing paper, and preferably equipped with two or three different pendulum weights and corresponding scales of different capacities, including scales with capacities of 0 to 1

<sup>1</sup> Under the standardization of the procedure Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

kg. (0 to 2.2 lb.) and 0 to 5 kg. (0 to 11 lb.). It shall conform to the requirements prescribed in Section 2 of the Tentative Method of Test for Tensile Breaking Strength of Paper and Paper Products (A.S.T.M. Designation: D 828).<sup>3</sup> It shall be calibrated as described in Section 3 of A.S.T.M. Method D 828.

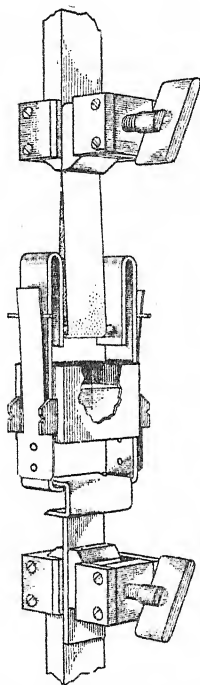


FIG. 1.—Wet Strength Device.

**METHOD A. FOR TISSUE, TOWEL, AND OTHER ABSORBENT PAPERS WHICH ARE EASILY DAMAGED BY HANDLING WHEN WET**

**Test Specimens**

3. The test specimens shall be cut from samples selected as prescribed in the Standard Method of Sampling Paper and Paper Products (A.S.T.M. Designation: D 585).<sup>3</sup> If desired and if possible, the specimens shall be cut in the two

principal directions of the sheet. Specimens shall be strips cut straight and parallel with smooth edges to within 0.1 mm. (0.004 in.) of the specified width. The test specimens shall be 12.7 mm. (0.5 in.), 15.0 mm. (0.59 in.), or 15.9 mm. (0.625 in.) in width and approximately 25 cm. (10 in.) in length.

**Procedure**

4. (a) Make the test in an atmosphere conditioned in accordance with the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>3</sup>

(b) Fasten the Finch wet strength device in the lower clamp of the tension testing machine so that the horizontal rod is placed at 90 deg. with the axis of the two clamps and parallel to the clamp faces and is otherwise symmetrically located with respect to the clamps. Adjust the position of the lower clamp so that the horizontal rod is about 7.6 cm. (3 in.) below the upper clamp. Move the liquid container to its lowest position, and almost fill it with the proper liquid. Unless otherwise required, the liquid shall be maintained at the temperature of the conditioned atmosphere,  $23 \pm 2$  C. ( $73 \pm 3.5$  F.). Wipe dry the horizontal rod and its supports, and thread the test specimen under the rod. Place the ends of the specimen together, remove the slack, and fasten them in the upper clamp of the tension testing machine. The specimen shall be centrally located with respect to the horizontal rod and the upper clamp. Raise the liquid container so that it locks in its uppermost position, immersing the looped end of the specimen to a depth of at least 19 mm. (0.75 in.).

(c) For easily saturated papers the immersion time shall be sufficient to develop their minimum strength which is produced by complete saturation. Determine this time by testing specimens

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

which have been immersed for progressively increasing periods. Select the shortest period resulting in a stable, low-strength value. For tissue, towel, and impregnating papers, the immersion time will be about 5 to 40 sec.

(d) At the end of the saturating period apply a tension load to the specimen at a rate conforming to that prescribed in Section 5 (c) of A.S.T.M. Method D 828, namely, that the rate of loading shall be adjusted so that the specimen breaks in not less than 5 nor more than 15 sec. Make the application of the first increments of load to the specimen very slowly, if possible, to minimize abnormal strains due to inertia effects.

(e) One half the load recorded when the test specimen breaks shall be taken as the wet tensile strength. At least ten specimens from each direction of the sheet shall be tested.

#### Report

5. The report shall include the following:

(1) Average, maximum, and minimum wet tensile strengths (to two significant figures) for each direction of the sheet, calculated to kilograms per 15-mm. width or to pounds per 1-in. width,

(2) Rate of travel of lower clamp,

(3) Kind of liquid used,

(4) Temperature of the liquid,

(5) Width and thickness of the test specimen, and

(6) Number of tests made.

**METHOD B. FOR WATER-RESISTANT PAPERS, SUCH AS PHOTOGRAPHIC, BLUE-PRINT, AND SPECIAL MAP PAPERS, WHICH CAN BE HANDLED WITHOUT DAMAGE AFTER IMMERSION**

#### Test Specimen

6. The test specimens shall be pre-

pared as prescribed in A.S.T.M. Method D 828.

#### Procedure

7. (a) The test specimens shall be immersed for the required time (Note) in the proper liquid. Unless otherwise required, the liquid shall be maintained at the temperature of the conditioning atmosphere,  $23 \pm 2$  C. ( $73 \pm 3.5$  F.).

**NOTE.**—The use requirement of the paper will establish the time of immersion which should be stipulated in the specifications for the paper. If the time is not specified, the specimens should be completely saturated by at least a 24-hr. immersion.

(b) At the end of the immersion time and before placing the specimen in the tension testing machine, place it flat and straight on a pad of four thicknesses of dry blotting paper and cover it with a similar pad. Roll a metal cylinder weighing  $500 \pm 5$  g. ( $17.5 \pm 0.25$  oz.) and having a diameter of about 5 cm. (2 in.) steadily over the top blotter, taking about 2 sec. for the operation.

(c) Immediately after blotting, place the test specimen in the tension testing machine and break the specimen as prescribed in Section 5 (c) of A.S.T.M. Method D 828.

(d) At least ten specimens from each direction of the sheet shall be tested.

#### Report

8. The report shall include the information prescribed in Section 5, and the following:

(1) Length of the specimen, and

(2) Wet tensile strength as a percentage of the dry tensile strength, if the paper is treated to maintain high wet strength.



## *Tentative Method of Test for* WATER VAPOR PERMEABILITY OF PAPER AND PAPERBOARD<sup>1</sup>



A.S.T.M. Designation: D 783 - 44 T

ISSUED, 1944.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method of test covers the procedure for determining the water vapor permeability of paper and paperboards under normal atmospheric conditions.<sup>3</sup> It is considered generally suitable also for other sheet materials up to 1 in. in thickness.

### Definition

2. Water vapor permeability is defined for this test as the weight of water transmitted per unit of time, per unit

area, measured while the specimen separates a dry atmosphere in a test cell from the standard atmosphere of 50 per cent relative humidity at a temperature of 73 F. on the other face.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Test Dish*.—An open mouthed cup or dish of such size and shape that it can be accommodated readily on the pan of an analytical balance. The area of the opening shall be as large as practicable, an area of at least 30 sq. cm. being preferred. The test dish shall be of such design that a wax seal can be made that will be impervious to leakage of water vapor and will define clearly the test area. Suitable designs for the dish with supporting rings and flanges are shown in Fig. 1. Other modifications of these designs may be made, providing the principle of preventing edge leakage by means of a complete wax seal is retained.

(b) *Template*.—A template for use in defining the test area and effecting the wax seal consisting of a circular metal

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1944.

<sup>3</sup> For further details on this method reference should be made to the following:

Allen Abrams and W. A. Chilson, *Paper Trade Journal*, Vol. 91, No. 18, October 30, 1930, p. 175; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 14, May, 1931, pp. 379-384.

Allen Abrams and G. J. Brabender, *Paper Trade Journal*, Vol. 102, No. 15, April 9, 1936, p. 32; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 19, June, 1936, pp. 121-130.

G. J. Brabender, *Paper Trade Journal*, Vol. 108, No. 4, January 26, 1939, p. 39; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 22, pp. 251 (1939).

F. T. Carson, *Miscellaneous Publication M 127*, Nat. Bureau Standards, August 5, 1937.

F. T. Carson, *Paper Trade Journal*, Vol. 107, No. 18, November 3, 1938, p. 59; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 20, pp. 90-102 (1937).

G. J. Brabender, *Paper Trade Journal*, Vol. 110, No. 18, May 2, 1940, p. 27; *Technical Association Papers*, Tech. Assn. Pulp and Paper Industry, Vol. 23, p. 358 (1940).

disk  $\frac{1}{8}$  in. in thickness with the edge beveled to an angle of about 45 deg. The diameter of the bottom (smaller) face of the template shall not be greater than the diameter of the effective opening of the cell in contact with the specimen.

(c) *Desiccant*.—A desiccant having a powerful affinity for water vapor and a high drying efficiency, that is, a low vapor pressure after absorbing a large

(d) *Wax*.—Wax for sealing the specimen to the test dish, made up of equal parts of crude beeswax and rosin. This wax shall cling tenaciously to surfaces and shall not be brittle at ordinary room temperature.

(e) *Petrolatum*.—Petrolatum for application to the beveled edge of the template in order to facilitate removal of the template after sealing the test speci-

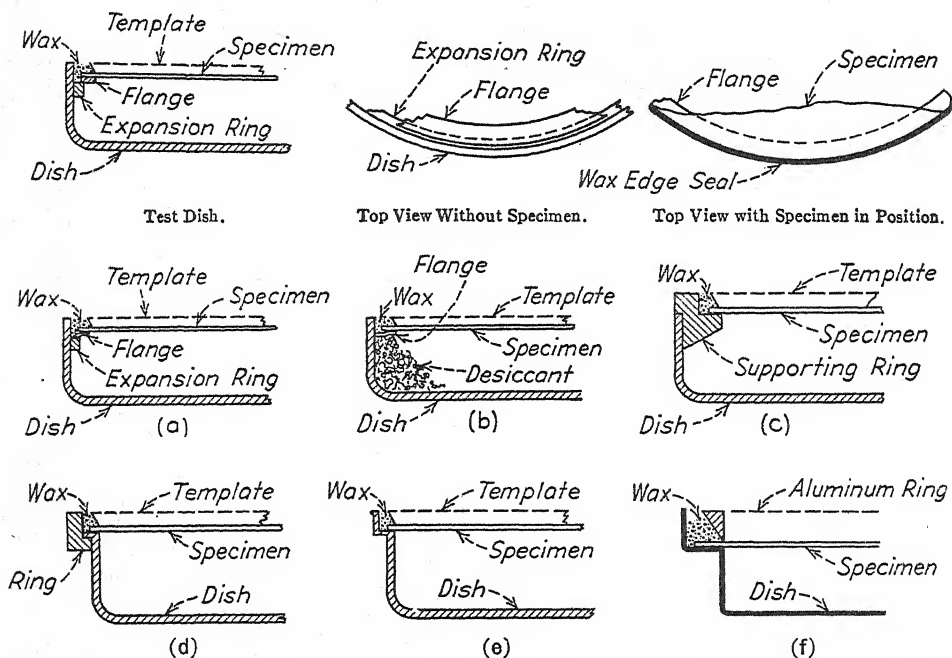


FIG. 1.—Several Types of Test Dishes.

amount of water. The desiccant shall remain essentially unchanged in physical condition and exert, while dry, no chemical or physical action, other than dehydration effects, on membrane materials with which it is in contact.<sup>4</sup> The desiccant used shall be in the form of small lumps, free from fines that will pass a No. 30 sieve.

<sup>4</sup> Anhydrous magnesium perchlorate made by the G. Frederick Smith Co., Columbus, Ohio, has been found satisfactory. This substance may be explosive at elevated temperature and should not be regenerated. Anhydrous calcium chloride is also suitable.

men to the dish. The petroleum jelly commonly used by druggists is suitable.

(f) *Balance*.—An analytical balance of 200-g. capacity, sensitive to 0.001 g.

(g) *Tare*.—A tare which shall be a blank specimen and dish assembly, similar to the test specimen assembly described in Section 4, except that no desiccant shall be contained in the dish. The purpose of the tare is to minimize in successive weighings the effects of extraneous influences, such as moisture absorption by dish, wax, dust deposits, and changes in

density of the confined air resulting from fluctuations in barometric pressure. The tare shall be subjected, along with the specimen assemblies, to the conditioning atmosphere.

(h) *Test Chamber*.—A test room or cabinet provided with conditioned air maintained at 50 per cent relative humidity at 73 F., and continuously circulated at a rate not less than 500 ft. per min. over the exposed surface of the specimens under test. A suitable grill or reticulated rack shall be provided in the test room or cabinet, capable of supporting the inverted specimens in a manner to afford free access of the circulating air.

### Test Specimens

4. Test specimens shall be representative of the material being tested. At least four specimens from each sample shall be tested, one half being attached to the cell with the wire side out, and the other half with the felt side out.

### Procedure

5. (a) Place sufficient desiccant inside the dish to cover the sheet area to a depth of at least 15 mm.

(b) For paper and paperboard  $\frac{1}{8}$  in. and less in thickness affix the test specimen to the dish as follows: Cut the test specimen so that its diameter is equal to that of the larger diameter of the template. Place the sheet over the aperture of the dish and center it as closely as possible on the supporting ring or flange. With the tip of the finger apply a thin film of petrolatum to the beveled edge of the template. Wipe off any petrolatum which may have been deposited on the smaller surface of the template. Center the template with the smaller surface down, exactly over the specimen and dish opening. Flow molten wax into the annular space surrounding the beveled edge of the template, using a

medicine dropper to dispense the molten wax. Remove the template from the sheet surface as soon as the wax has cooled and solidified.

(c) For fiberboards over  $\frac{1}{8}$  in. in thickness, modify the method of preparing the test assembly (Paragraph (b)) as follows: To prevent edge leakage, rotate the edge of the test specimen in molten wax of such fluidity that it does not penetrate into the edge more than  $\frac{1}{16}$  in., suspend inside the dish to a distance of about one half the thickness of the specimen, and run molten wax around the edge of the specimen to seal it to the dish.

(d) Weigh the assembly on the analytical balance to 0.001 g., using the tare. Place the dish on the rack inside the test room or cabinet in an inverted position so that the layer of desiccant is in direct contact and evenly distributed over the inner face of the test sheet, and so that free access of the conditioned circulating air is provided on the exposed surface of the sheet.

(e) Make successive weighings of the assembly at suitable intervals until a constant rate of gain is attained. For papers that are relatively pervious, the weighings should be frequent enough to complete the test before drops of liquid agglomerate are formed in the desiccant or caking of the desiccant occurs. These conditions will be indicated by a drift from the constant rate of gain after which no readings should be recorded. Plot the weight gain against time. The slope of the resulting curve will furnish a measure of the water-vapor permeability.

### Report

6. (a) The water-vapor permeability shall be reported as grams per square meter or ounces per square yard per 24 hr. at 73 F. The calculation should be made for the period of constant rate of gain (Note).

**NOTE: Example.**—In a test run for 190 hr. on an exposed area of 55 sq. cm. (0.0055 sq. m.), it was found that the rate of gain was substantially constant after 18 hr. and that during the subsequent 172 hr., the moisture absorbed by the desiccant was 67 mg.; the water-vapor permeability is then  $\frac{0.067 \times 24}{0.0055 \times 172} = 1.70$  g. of moisture passing through 1 sq. m. of the material every 24 hr. at 73 F. from 50 per cent relative humidity on one face to desiccant in contact with the other. For purpose of calculation: ounces per 100 sq. yd. = grams per sq. m.  $\times 2.95$ .

(b) The permeability values shall be reported for each side of the sheet separately, the side designated in each case being that which faced the higher humidity in the test. A suitable code designation should be used to distinguish

between the two sides of the material, such as Side I and Side II; or when there is an obvious difference between the two sides, such as Side I, waxed, and Side II, unwaxed. The report shall state what desiccant was used.

#### Reproducibility

7. Duplicate determinations should check within 10 per cent, depending largely on the variation in the sheet material itself. As the spread between very permeable sheets and those having a low permeability is at least a thousand-fold, a precision of 10 per cent on a given material will establish it quite definitely in the scale of permeabilities normally encountered in sheet materials.

## *Tentative Method of Test for*

# WATER VAPOR PERMEABILITY OF PAPER AND OTHER SHEET MATERIALS AT ELEVATED TEMPERATURE AND HUMIDITY<sup>1</sup>



A.S.T.M. Designation: D 830 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers the procedure for determining the permeability to water vapor of paper and other sheet materials at high temperature and humidity. The influence of temperature and humidity on the water vapor transmission of most sheet materials is not linear so that test results obtained under the standard atmospheric conditions prescribed in the Tentative Method of Test for Water Vapor Permeability of Paper and Paperboard (A.S.T.M. Designation: D 783)<sup>3</sup> cannot be extrapolated to apply to excessively warm, damp atmospheric conditions. This method provides a means of predicting the permeability that will occur under such conditions.

### Definition

2. Water vapor permeability is defined for the purpose of this test as the

weight of water transmitted per unit of time, per unit area, measured while the specimen separates a dry atmosphere in a test cell from an atmosphere of 90 per cent relative humidity at a temperature of 100 F. on the other face.

### Apparatus

3. The apparatus shall consist of the following:

(a) *Test Dish*.—An open-mouth cup or dish of such size and shape that it can be accommodated readily on the pan of an analytical balance. The opening should be as large as practical, preferably with an area of at least 30 sq. cm. The test dish shall be of such design that a wax seal can be made that will be impervious to leakage of water vapor and will define clearly the test area. Suitable designs for the dish with supporting rings and flanges are shown in Fig. 1. Other modifications of these designs may be made, provided the principle of preventing edge leakage by means of a complete wax seal is retained.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(b) *Template*.—A template for use in defining the test area and effecting the wax seal consisting of a circular metal disk  $\frac{1}{8}$  in. in thickness, with the edge beveled to an angle of about 45 deg. The diameter of the bottom face of the template shall not be greater than the diameter of the effective opening of the cell in contact with the specimen.

(c) *Desiccant*.—A desiccant such as anhydrous calcium chloride or mag-

small lumps, free from fines that will pass a No. 30 (590-micron) sieve.

(d) *Wax*.—Nonabsorbent wax for sealing the specimen to the testing dish, made up of 60 per cent amorphous wax,<sup>5</sup> and 40 per cent refined crystalline paraffin wax. The mixture shall cling tenaciously to surfaces, and shall not be brittle at room temperatures. It may be tested for vapor absorption by exposing a heavy coating on a glass plate

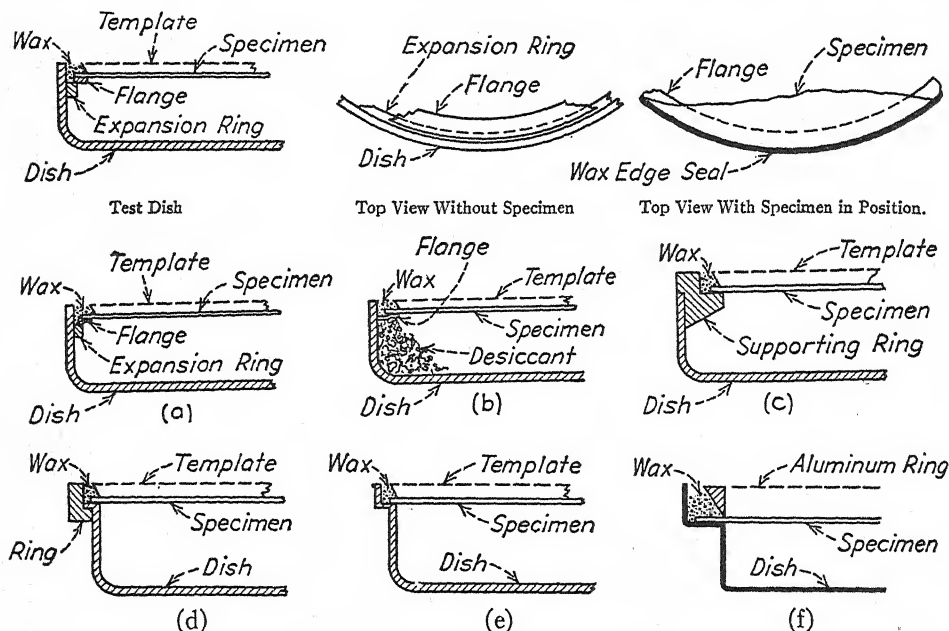


FIG. 1.—Several Types of Test Dishes.

nesium perchlorate, with a powerful affinity for water vapor and a high drying efficiency, that is, a low vapor pressure after absorbing a large amount of water. The desiccant shall remain essentially unchanged in physical condition and exert, while dry, no chemical or physical action other than dehydration effects on membrane materials with which it is in contact.<sup>4</sup> The desiccant used shall be in the form of

to the test atmosphere and checking it for changes in weight.

(e) *Petrolatum*.—Petrolatum for application to the beveled edge of the template in order to facilitate removal of the template after sealing the test specimen to the dish. The petroleum jelly commonly obtainable from druggists is suitable.

(f) *Balance*.—An analytical balance of 200-g. capacity, sensitive to 0.001 g.

(g) *Cabinet*.—A cabinet wherein cir-

<sup>4</sup> Anhydrous magnesium perchlorate made by the G. Frederick Smith Co., Columbus, Ohio, has been found satisfactory. This substance may be explosive at elevated temperature and should not be regenerated. Anhydrous calcium chloride is also suitable.

<sup>5</sup> Grades Nos. 2300, 2305, and 2310 of the Socony Vacuum Oil Co. are satisfactory for this purpose.

culating air shall be maintained at  $100 \pm 1$  F. and  $90 \pm 2$  per cent relative humidity. Suitable racks or grills shall be provided to support the test specimens in the conditioned atmosphere which shall be circulated continuously.

(h) *Covers*.—Weighing covers for the test specimens shall be provided if the design of the cabinet is such that it is necessary to remove the specimens from the cabinet for weighing. These covers should consist of circular disks of aluminum  $\frac{1}{8}$  to  $\frac{3}{8}$  in. in thickness, each with a suitable knob in the center for lifting. The cover shall fit over the test specimen when assembled and make contact with the inside beveled surface of the wax seal at or just above the plane of the specimen. All sharp edges shall be relieved to avoid removing any wax. Each cover shall be numbered or otherwise identified to facilitate its always being used with the same dish.

### Test Specimens

4. The test specimens shall be representative of the material. At least two specimens shall be prepared for each type of test required. The different types of tests to be made as specified include: (1) flat specimens with designated side exposed to the high humidity, and (2) creased specimens, as specified, with designated side exposed to the high humidity.

### Procedure

5. (a) Check the relative humidity and temperature of the test atmosphere. This is a difficult operation with some cabinets. Depending on the design of the cabinet this may be accomplished by using either a wet and dry-bulb thermometer, or a wet and dry contact thermocouple (Note 1), or more approximately, by checking the weight stability of appropriate saturated salt solutions (Note 2) exposed in crystallizing dishes to the test atmosphere.

NOTE 1.—Air at 100 F. and 90 per cent relative humidity has a wet-bulb temperature of 97.2 F.

NOTE 2.—Saturated solutions of barium chloride, ammonium dihydrogen phosphate, and potassium sulfate are in weight equilibrium with 88 per cent, 91.5 per cent, and 96 per cent humidity, respectively.

(b) Place sufficient desiccant inside the dish to cover the sheet area to a depth of at least 15 mm. Cut the test specimen so that its diameter is equal to that of the larger diameter of the template. Place the sheet over the aperture of the dish and center it as closely as possible on the supporting ring or flange. With the tip of the finger apply a thin film of petrolatum to the beveled edge of the template. Wipe off any petrolatum that may have been deposited on the smaller surface of the template. Center the template with the smaller surface down, exactly over the specimen and dish opening. Flow molten wax into the annular space surrounding the beveled edge of the template. Remove the template from the sheet surface as soon as the wax has cooled and solidified. Remove loose wax and check seal for flaws.

(c) To use the template shown in Fig. 1 (b), fill the desiccant into a suitable tray, center the specimen on the template, and place the tray on the specimen. Place the inverted dish in position, make the wax seal as described in Paragraph (b), and invert the assembly.

(d) To use the type of test dish shown in Fig. 1 (c), seal one specimen in position, then invert the assembly, practically fill the desiccant space with the desiccant, taking care not to leave any powder on the sealing ledge, and seal the other specimen in position.

(e) Prepare the desired number of specimen assemblies for test and number each test dish. Place them on the shelves of the test cabinet at 100 F. and 90 per cent relative humidity for a



period of at least 16 hr. When the conditioning period is judged to be completed this may take several days for very impermeable specimens), make successive weighings daily until a constant rate of gain is attained. This is best accomplished by plotting the cumulative gain in weight in milligrams of each assembly against the elapsed time in the cabinet, until at least three, and preferably four, of the resulting points are in a straight line.

(f) If the design of the cabinet is such that weighings cannot be made without removing the specimens, open the cabinet and cover each dish with its correspondingly numbered cover, as it becomes accessible in the cabinet. Weigh each numbered dish and cover, in numerical sequence, on an analytical balance to the nearest 1 mg. Record the weight of each and replace them in the shelf of the cabinet, removing the covers as they are returned. Covers should be kept in a dust-free place, each preferably resting flat on three pointed supports. Note the time, and allow the specimen assemblies to remain in the cabinet for a suitable interval (approximately 24 hr. or longer).

(g) At the end of this period, note the time and again weigh to the nearest 1 mg. each assembly with its corresponding cover in position in the same sequence as before. Again replace them in the cabinet, removing the covers as they are returned, and note the time. Make successive weighings of the assemblies at suitable intervals until a constant rate of gain in weight is attained.

(h) It is important that the assemblies be weighed in numerical sequence and

replaced in the cabinet in the same order each time the weighings are made. Precisely the same routine of weighing and replacing the test assemblies shall be followed in detail at each interval to minimize possible sources of error.

### Calculation

6. The water vapor permeability shall be calculated as grams per square meter per 24 hr. at 100 F. and for a moisture gradient as defined in this method. The calculation shall be made for the period of constant rate of gain or loss.

NOTE: *Example.*—In a test run of 120 hr. on an exposed area of 55 sq. cm. (0.0055 sq.m.) it was found that the rate of gain was substantially constant after 48 hr., and that the increase in weight for the next 72 hr. was 25 mg. The water vapor permeability was:

$$\frac{0.025 \times 24}{0.0055 \times 72} = 1.5 \text{ g. per sq. m. per 24 hr.}$$

### Report

7. The report shall include the following:

(1) The average, maximum, and minimum values for each type of test made (to two significant figures),

(2) The type of test, for example, "this test made on uncreased specimens with side I exposed to the high humidity," or "this test made on creased specimens with coated side exposed to the high humidity," and

(3) Desiccant used.

### Reproducibility

8. Using different pieces of apparatus in different laboratories, the results obtained by this method should be within 20 per cent of the mean, or 0.2 g. per sq. m. per day, whichever is the larger.

## *Tentative Method of Test for* DEGREE OF WET CURL OF PAPER<sup>1</sup>



A.S.T.M. Designation: D 826 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method of test covers the procedure for determining the maximum curvature developed and the time required to reach this maximum curvature by means of bringing one side of the test specimen in contact with water. The relative tendency of certain papers, especially printing papers, to curl under normal conditions of usage (Note) is indicated by the maximum curvature.<sup>3,4</sup> The time required to reach maximum curvature is a measure of the relative degree of sizing, such as secured with rosin.<sup>5</sup>

NOTE.—The factors out of which curling troubles grow are many and complex. It is therefore necessary to use caution in interpreting curling data obtained by this test in connection with certain types of curling troubles. A high

wet curl test does not necessarily mean that the paper will, in every case, curl in practice.

### Apparatus

2. (a) *Curl Tester*.—The curl tester (Fig. 1) consists of a float near the center of which is a rectangular wetting aperture approximately 2 in. in length (perpendicular to plane of figure) and having a width,  $L$ , which is adjustable from 0 to about 1 in. A stop,  $H$ , is provided to hold one end of the test specimen at a fixed angle,  $F$ . A scale, with stippled surface, graduated in degrees, is fastened to the float to indicate the movement of the pointed end of the test specimen.

NOTE.—The curl sizing tester is available for testing both the degree of curl and the rate of curl. For the latter purpose, the apparatus contains a built-in stop watch which starts automatically when the specimen is lowered to the water.

(b) *Stop Watch*.—A stop watch or other suitable timing device.

(c) *Water*.—A vessel containing water at  $23 \pm 2$  C. Distilled water is preferable, but drinking water of reasonable purity may be used.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-6 on Paper and Paper Products.

<sup>2</sup> Accepted by Committee E-10 on Standards, June 27, 1945.

<sup>3</sup> F. T. Carson and V. Worthington, "Simplified Curl Tester for Paper," *Paper Industry and Paper World*, Vol. 22, No. 3, June, 1940, pp. 246-247.

<sup>4</sup> F. T. Carson and V. Worthington, "Measuring the Degree of Curl of Paper," *Journal of Research*, Nat. Bureau Standards, Vol. 30, No. 2, February, 1943, p. 113. (*Research Paper RP1522*.)

<sup>5</sup> F. T. Carson, "New Sizing Tester for Paper," *Paper Trade Journal*, Vol. 79, No. 17, October 23, 1924, pp. 44-45.

### Test Specimens

3. The test specimens shall be cut in the shape of right trapezoids with an altitude of  $1\frac{1}{2}$  in. in the machine direction and bases of  $1\frac{1}{2}$  in. and  $2\frac{3}{4}$  in. extending in the cross direction.<sup>3</sup> Specimens shall be conditioned before testing as prescribed in the Standard Method of Conditioning Paper and Paper Products for Testing (A.S.T.M. Designation: D 685).<sup>6</sup>

### Procedure

4. (a) Make the tests in an atmosphere conditioned as prescribed in the A.S.T.M. Method D 685.

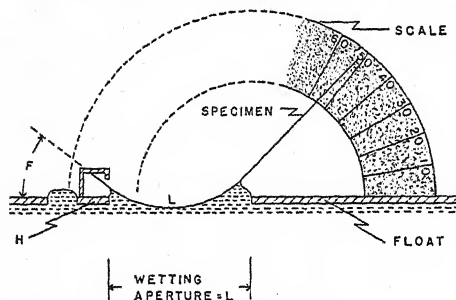


FIG. 1.—Curl Tester.

(b) *Degree of Curl.*—Place the float on the water; place the square end of the trapezoidal test specimen under the stop with the longest, pointed side adjacent to the scale, and allow the specimen to fall across the aperture and become wet over that area. Read the scale angle reached by the point of the specimen at the instant the direction of curling reverses. If this angle is less than 30 or more than 60 deg., adjust the width of the aperture until the angle lies within the desired range. In gene-

ral, if the curve of the specimen departs noticeably from a circular cylindrical surface, reduce the width, in the cross direction, of the wetted portion of the specimen until the curve is substantially circular. Test at least five specimens in this way from each side of the paper, making sure that the top of the float is dry before each test. Add the average scale angle to the fixed angle,  $F$  (approximately 30 deg.) which the square end of the specimen makes with the horizontal. Divide this sum by the width in centimeters of the aperture used (length of arc of wetted, curled portion of specimen in cross direction). The result is the maximum curvature in degrees per centimeter of arc.

(c) *Rate of Curl.*—Place the test specimen in the apparatus as described in Paragraph (b). With the stop watch determine the time from contact of the specimen with the water until the maximum curvature is reached, that is, until the point of the specimen reverses its direction of movement. For this test it is preferable to adjust the width of the aperture so that maximum curvature is reached near the middle of the scale. Test at least five specimens from each side of the paper.

### Report

5. The report shall include the following:

(1) *Degree of Curl.*—The average value and the range of maximum curvature, in degrees per centimeter of aperture width, and

(2) *Rate of Curl.*—The average value and the range of time in seconds to reach maximum curvature.

<sup>6</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

# *Tentative Method of*

## COMPRESSION TEST FOR SHIPPING CONTAINERS<sup>1</sup>



A.S.T.M. Designation: D 642 - 45 T

ISSUED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method covers two procedures for making compression tests on shipping containers, usually on the containers without contents, as follows:

*Procedure A* for measuring the ability of the container to resist external compressive loads applied to its faces, and

*Procedure B* for measuring the ability of the container to resist external compressive loads applied to diagonally opposite edges or corners.

(b) Either method of applying load may be used to compare the characteristics of a given design of container with a standard or to compare the characteristics of containers differing in construction. These procedures are suitable for testing boxes, crates, barrels, drums, kegs, and pails made of metal, wood, fiberboard, and combinations of these materials.

### Apparatus

2. The apparatus shall consist of the following:

(a) *Testing Machine*.—A compression testing machine of accepted design and capacity. It shall be calibrated in accordance with the Standard Methods of Verification of Testing Machines (A.S.T.M. Designation: E 4).<sup>3</sup> If the machine is not equipped with an autographic recording device that records load and deformation, auxiliary equipment shall be provided to measure deformation.

(b) *Sealing Equipment for Fiberboard Boxes*.—Suitable facilities, including sealing boards and proper adhesive, for sealing both the top and bottom flaps of box specimens without bracing material within the boxes that will give false results as to compressive strength.

(c) *Conditioning Apparatus*.—Adequate facilities for conditioning test specimens at proper humidity and temperature prior to test in accordance with the requirements of the specifications covering the containers to be tested.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-10 on Shipping Containers.

<sup>2</sup> Accepted by Committee E-10 on Standards, August 27, 1945.

Prior to its present publications as tentative, this method was published as tentative from 1941 to 1943. It was adopted in 1943 and published as standard from 1943 to 1945 when it was revised and republished as tentative.

<sup>3</sup> Appears in this publication, see Contents in Numerical Sequence of A.S.T.M. Designations at front of book.

(d) *Miscellaneous Equipment*.—Dry-iron oven, scales, knife, saws, etc., for use in determination of the moisture content or for making other supplementary tests of the materials from which the containers are made.

#### Test Specimens and Number of Tests

3. (a) The containers being tested shall be complete in all respects with any and all interior packing. Tests shall be made on containers with or without contents as prescribed. Packed containers shall be closed and strapped in the same manner as will be used in preparing them for shipment.

(b) Performance normally should be based on tests of not less than three representative specimens of a given size and type of container. No specimen shall be used for more than one type of test.

#### Sealing Fiberboard Containers

4. (a) The box specimen shall be sealed so as to avoid distortions that may affect its load-bearing ability. The method of preparing the test specimen as described in Paragraphs (b) to (e) will accomplish this, but any method that will produce the same result may be used.

(b) The box specimens shall be sealed as follows: The box blank shall be squared up and each flap in turn bent backward 180 deg. on the score line and then forward 270 deg. to the normal closure position. Starting with either the top or bottom of the box, the short or inner flaps shall be closed first and each of them shall be given a uniform application of adhesive, such as silicate of soda. Then the long or outer flaps shall be closed onto the glued surface of the inner flaps. The adhesive shall be kept at least  $\frac{1}{2}$  in. away from the score lines. The closure shall be placed flat on a solid level surface and a flat,

rigid board slightly smaller than the closure area placed on the inner surface of the closure, and weight applied to the board by one of the following means:

(1) Placing weights on the board, or

(2) Inserting a screw hook through a slot in the work bench and through the mid-point of the junction of the outer flaps into the board, from which screw hook a weight is suspended, or

(3) Making a hole in the center of the board through which a carriage bolt is passed down through the junction of the flaps and through a slot in the bench to receive a large wing nut which tightens the board in place, thus transmitting pressure to the glued surfaces.

(c) When the first closure glue has set sufficiently to allow handling, the boards and weight or bolt shall be removed.

(d) The other face of the box specimen shall be sealed as follows: A board similar in size to that used for the first closure shall be suspended in the opening of the box. A carriage bolt or screw hook shall be placed so that it extends upward through the center of the board. The short or inner flaps shall be flexed first outward and then inward as described in Paragraph (b), and then brought to rest on the board, and given a coating of glue. Then the longer or outer flaps shall be flexed outward and inward and brought to rest on the glued surfaces. Pressure shall then be applied to this glued joint by one of the following means:

(1) Inverting the box with the screw hook extending through a slot in the work bench and suspending from the hook sufficient weight to ensure firm contact, or

(2) Inverting the box with a bolt extending through a slot and tightening a wing nut on the bolt to exert pressure, or

(3) Slipping a second board down over the bolt, and tightening the nut to draw the two boards together, thus holding the glued joint while it sets.  
(e) When the glue has set, the clamp-

because it is of such a size as to prevent wedging.

#### Conditioning

5. Depending on the purpose of the

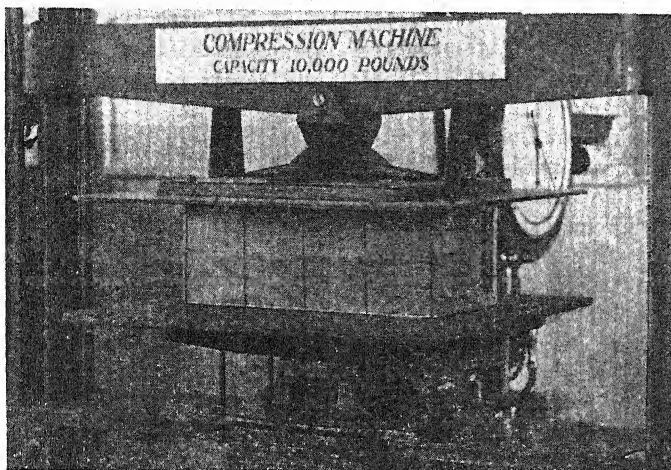


FIG. 1.—Compression Applied Top-to-Bottom.

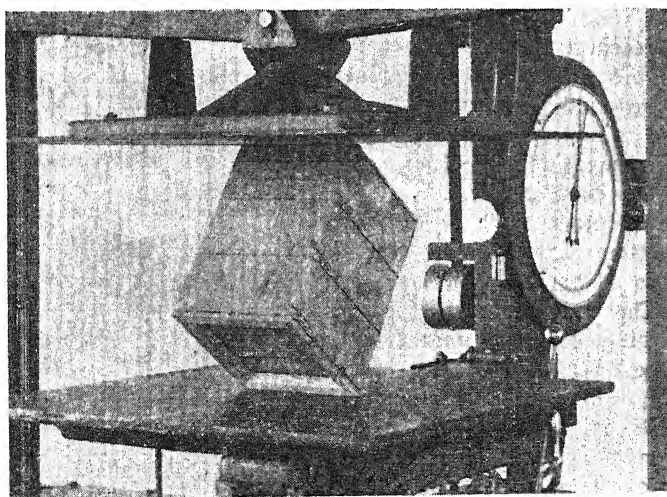


FIG. 2.—Compression Applied to Diagonally Opposite Edges.

ing device shall be released, thus obtaining a completely sealed box without contents, except for the inner sealing board, which falls loose from the flaps and will not offer any support to the box

tests, the containers may be conditioned prior to the compression test by either water immersion, exposure to water spray, or exposure to fixed conditions of air temperature or humidity.



### Moisture Content

6. When determination of moisture content is required, the following procedures shall be used:

(a) *Fiberboard*.—The moisture content of fiberboard at the time of test shall be determined in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (A.S.T.M. Designation: D 644).<sup>3</sup>

(b) *Wood*.—The moisture content of wood at the time of test shall be determined by selecting duplicate samples from different parts of the container, immediately following the test, of approximately 2 cu. in. each, weighing to the nearest 0.1 g., drying to constant weight at 100 to 105 C. (212 to 221 F.), and reweighing. The moisture content shall be expressed as a percentage of the oven-dry weight of the specimen.

### Procedure A

7. (a) Procedure A shall be used for measuring the ability of the container to resist external compressive loads applied to its faces. The compression test shall be made top-to-bottom, end-to-end, or side-to-side.

(b) The specimen shall be centered on the bottom platen of the testing machine, so as not to incur eccentric loading. The top platen shall then be lowered until it comes in contact with the specimen (see Fig. 1). An initial pressure of 50 lb. shall then be applied to insure a definite contact between the specimen and the platens. The distance between the platens at this time shall be recorded as zero deformation. With this 50-lb. load on the specimen, the autographic load - deformation recorder pen shall be set at zero deformation. If the testing machine is not fitted with an autographic recorder, the test load shall be recorded for every 0.1-in. or

$\frac{1}{8}$ -in. deformation of the container. The load shall be applied with a continuous motion of the movable head of the testing machine, at a speed of  $\frac{1}{2} \pm \frac{1}{4}$  in. per min., until failure and maximum load or either has been reached.

(c) For each type of loading, critical points shall have been established and the compressive load at these critical

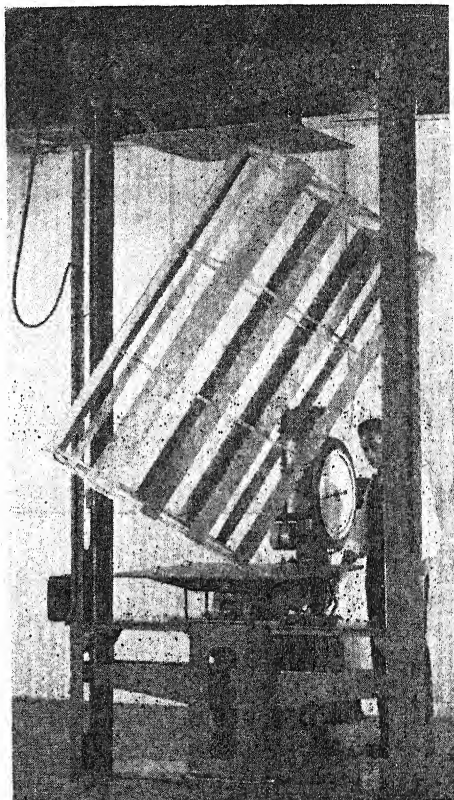


FIG. 3.—Compression Applied to Diagonally Opposite Corners.

deformations shall be recorded, together with the maximum load and deformation.

### Procedure B

8. Procedure B shall be used for measuring the ability of the container to resist external compressive loads



applied to diagonally opposite edges or corners. This test shall be made as described in Section 7, except that the container shall be centered in the machine on diagonally opposite edges (Fig. 2) or diagonally opposite corners (Fig. 3). The upper platen shall be fixed; that is, not equipped with a swivel joint.

### Report

9. The report shall include the following:

(1) Dimensions of the container under test; its complete structural specifications; kind of material; description and specifications for blocking and cushioning, if used; spacing, size, and kind of fasteners; method of closing and

strapping; and the net and gross weights.

(2) Description of the contents of the container, unless it is tested empty.

(3) A detailed record of the test on each container, including damage to the container and contents, together with any observations which may assist in correctly interpreting the results or aid in improving the design of the container.

(4) A graph or table showing the load-deformation relations for each test.

(5) The method, if any, of conditioning the container; the moisture content, if determined, of the wood, plywood or fiberboard; and the results of any supplementary tests of the materials from which the container is made.

(6) A statement to the effect that all tests were made in full compliance with this method.

# *Tentative Method of*

## DROP TEST FOR SHIPPING CONTAINERS<sup>1</sup>



A.S.T.M. Designation: D 775 - 45 T

ISSUED, 1944; REVISED, 1945.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method covers two procedures for making drop tests on loaded shipping containers, as follows:

*Procedure A* for measuring the ability of the container and inside packing materials to provide protection to its contents, and

*Procedure B* for measuring the ability of the container to withstand rough handling, and for providing information useful in improving the design of the container.

(b) Either procedure may be used to compare the characteristics of a given design of container with a standard or to compare the characteristics of containers differing in construction. These procedures are suitable for testing boxes, crates, barrels, drums, kegs, and pails made of metal, wood, fiberboard, or combinations of these materials.

(c) The procedures prescribed in this method are particularly suitable for containers that normally will be handled by manual methods in shipment. Con-

tainers having a gross weight of about 300 lb. or more, or containers of large size, may be more satisfactorily tested on the incline-impact tester.<sup>3</sup>

### Apparatus

2. The apparatus shall consist of the following:

(a) *Drop Test Apparatus*.—Any suitable apparatus may be used that conforms to the following requirements:

(1) Permits accurate prepositioning of the container to assure a true fall and impact at the exact places and in the direction desired,

(2) Permits accurate and convenient control of the height of drop,

(3) Facilitates handling and elevation of the containers, particularly when weights are in higher brackets,

(4) Utilizes lifting devices that will not damage the containers,

(5) Permits an absolutely free, unobstructed fall,

(6) Provides for variations in height of drops within limits of anticipated requirements, and

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-10 on Shipping Containers.

<sup>2</sup> Revision accepted by Committee E-10 on Standards, August 27, 1945.

<sup>3</sup> Tentative Method of Incline Impact Test for Shipping Containers (A.S.T.M. Designation: D 880), see p. 1271.

(7) Provides a solid surface of concrete, stone, or steel of sufficient mass to absorb all shock without deflection.

(b) *Conditioning Apparatus*.—Adequate facilities for conditioning test specimens at proper humidity and temperature prior to test in accordance with

tested in accordance with Procedure A, it shall be packed with the actual contents for which it was designed. When Procedure B is used, the container may be packed with either the actual contents or with a dummy load simulating these contents. Regardless of which procedure is used, the container shall be closed and

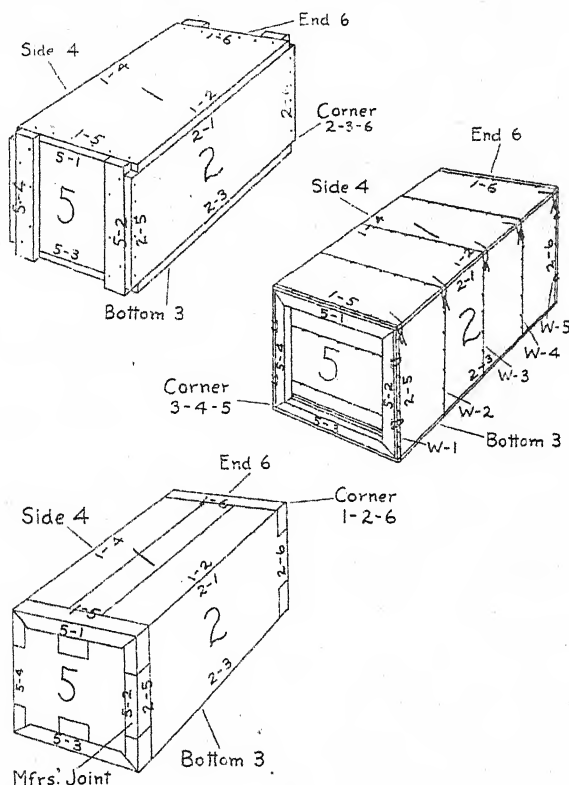


FIG. 1.—Diagram Showing Identification of Faces, Edges, and Corners of Containers.

the requirements of the specifications covering the containers to be tested.

(c) *Miscellaneous Equipment*.—Drying oven, scales, knife, saws, etc. for use in determination of the moisture content or for making other supplementary tests of the materials from which the containers are made.

#### Test Specimens and Number of Tests

3. (a) When the container is to be

strapped in the same manner as will be used in preparing it for shipment.

(b) The procedure for the identification of the faces, edges, and corners of containers shall be as follows (see Fig. 1): Facing one end of the container, with the manufacturer's joint on the observer's right if the container is fiberboard, designate the top of the container as 1, the right side as 2, the bottom as 3, the left side as 4, the near end as 5, and

the far end as 6. Identify the edges by the numbers of the two faces that form that edge; for example, 1-2 identifies the edge formed by the top and the right side, and 2-5 the edge formed by the right side and the near end. (The latter is the edge having the manufacturer's joint, in fiberboard containers.) Identify the corners by the numbers of the three faces that meet to form that corner; for example, 1-2-5 identifies the corner where the top, the right side, and the near end meet.

(c) Performance shall be based on tests of not less than five representative specimens of a given size and type of container.

### Conditioning

4. Depending on the purpose of the tests, the containers may be conditioned prior to the drop test by either water immersion, exposure to water spray, or exposure to fixed conditions of air temperature or humidity.

### Moisture Content

5. When determination of moisture content is required, the following procedures shall be used:

(a) *Fiberboard*.—The moisture content of fiberboard at the time of test shall be determined in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and Fiberboard Containers (A.S.T.M. Designation: D 644).<sup>4</sup>

(b) *Wood*.—The moisture content of wood at the time of test shall be determined by selecting duplicate samples from different parts of the container, immediately following the test, of approximately 2 cu. in. each, weighing to the nearest 0.1 g., drying to constant weight at 100 to 105 C. (212 to 221 F.), and reweighing. The moisture content shall be expressed as a percentage of the oven-dry weight of the specimen.

### General Procedure

6. (a) Containers may be dropped on a corner, a face, or an edge, or on each in a specified sequence. Before dropping on a corner or edge, the container shall be positioned with the center of gravity of the container and contents over the point or edge of impact. In the edgewise drop test, the edge on which the container is to drop shall be positioned in a horizontal line, parallel with the floor or base on which the container drops. In the flatwise drop test the face on which the container is to fall shall be positioned in a parallel plane with the floor. When the table-top drop test apparatus (see Appendix) is used, the container shall be placed in the center of the table and dropped by releasing the trap doors, which fall away quickly, allowing the container to fall vertically and freely to the floor or base. When the hoisting apparatus (see Appendix) is used, the container shall be properly lined up and the release hook or latch tripped, allowing the container to fall vertically and freely to the floor or base.

(b) *Height of Drop*.—The height from which containers shall be dropped will depend upon the purpose of the test. In some instances the height and number of drops will be prescribed in the specifications for the container.

### Procedure A

7. (a) Procedure A shall be used for measuring the ability of a container to provide protection to its contents. In this procedure, the drop test includes cornerwise, edgewise, and flatwise drops, with the exception that if the container is long and narrow, the flatwise drops on the long faces may be omitted.

(b) The container and contents shall be dropped from the prescribed height (Section 6(b)) in the following sequence, which constitutes the first cycle of 10 drops:

(1) A corner drop on the 5-1-2 corner,

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

(2) An edge drop on the shortest edge radiating from that corner,

(3) An edge drop on the next shortest edge radiating from that corner,

(4) An edge drop on the longest edge radiating from that corner,

(5) A flatwise drop on one of the smallest faces,

(6) A flatwise drop on the opposite smallest face,

(7) A flatwise drop on one of the medium faces,

(8) A flatwise drop on the opposite medium face,

(9) A flatwise drop on one of the largest faces, and

(10) A flatwise drop on the opposite large face.

(c) The second cycle of 10 drops shall be started with a drop on the 6-3-4 corner, which is diagonally opposite to the corner on which the first drop was made, and completed in the sequence prescribed in Paragraph (b).

(d) When additional cycles of 10 drops each are necessary, they shall be started at the following corners and completed in the sequence prescribed in Paragraph (b):

Cycle	Corner
Third.....	5-2-3
Fourth.....	6-1-4
Fifth.....	5-3-4
Sixth.....	6-1-2
Seventh.....	5-1-4
Eighth.....	6-2-3

(e) The test shall be continued until a specified predetermined number of drops, estimated to be equivalent to rough handling in actual service, have been given the container, at which time the container shall be opened and the contents inspected.

## Procedure B

8. (a) Procedure B shall be used for measuring the ability of the container to withstand rough handling. In this

procedure the drop test generally consists only of cornerwise drops.

(b) The container and contents shall be dropped cornerwise from the prescribed height (Section 6(b)) on the floor or base in the following sequence of drops: 5-1-2, 6-3-4, 5-2-3, 6-1-4, 5-3-4, 6-1-2, 5-1-4, and 6-2-3. These eight cornerwise drops shall constitute one cycle. Additional cycles shall be made at increased heights until failure of the container results. Failure, depending upon the purpose of the test, may be considered to have occurred when: (a) the contents are exposed, or (b) the contents spill from the container, or (c) one edge breaks open along its entire length, or (d) some other predetermined type of damage has occurred.

## Report

9. The report shall include the following:

(1) Dimensions of the container under test; its complete structural specifications; kind of material; description and specifications for blocking and cushioning, if used; spacing, size, and kind of fasteners; method of closing and strapping, if any; and the net and gross weights.

(2) Description of the contents of the container under test.

(3) A detailed record of test on each container, including damage to the container and contents, together with any other observation which may assist in correctly interpreting the results or aid in improving the design of the container or the method of packing, blocking, or bracing.

(4) The method, if any, of conditioning the container; the moisture content, if determined, of the wood, plywood, or fiberboard; and the results of supplementary tests of the materials from which the container is made.

(5) A statement to the effect that all tests were made in full compliance with this method.



# *Tentative Method of*

## INCLINE IMPACT TEST FOR SHIPPING CONTAINERS<sup>1</sup>



A.S.T.M. Designation: D 880 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. (a) This method covers two procedures for making incline impact tests on loaded shipping containers, as follows:

*Procedure A*, to determine the ability of a container to withstand impact stresses, and

*Procedure B*, to determine the ability of a container or interior packing, or both, to provide protection to the contents, when subjected to impact stresses.

(b) Either procedure may be used to obtain average results permitting a comparison of different designs of containers of the same size and carrying the same load. These procedures are suitable for testing various types of containers such as boxes, crates, barrels, drums, kegs, or pails made of metal, wood, fiberboard, or combinations of these materials.

(c) The procedures prescribed in this method are particularly suitable for testing large or heavily loaded containers.

### Apparatus

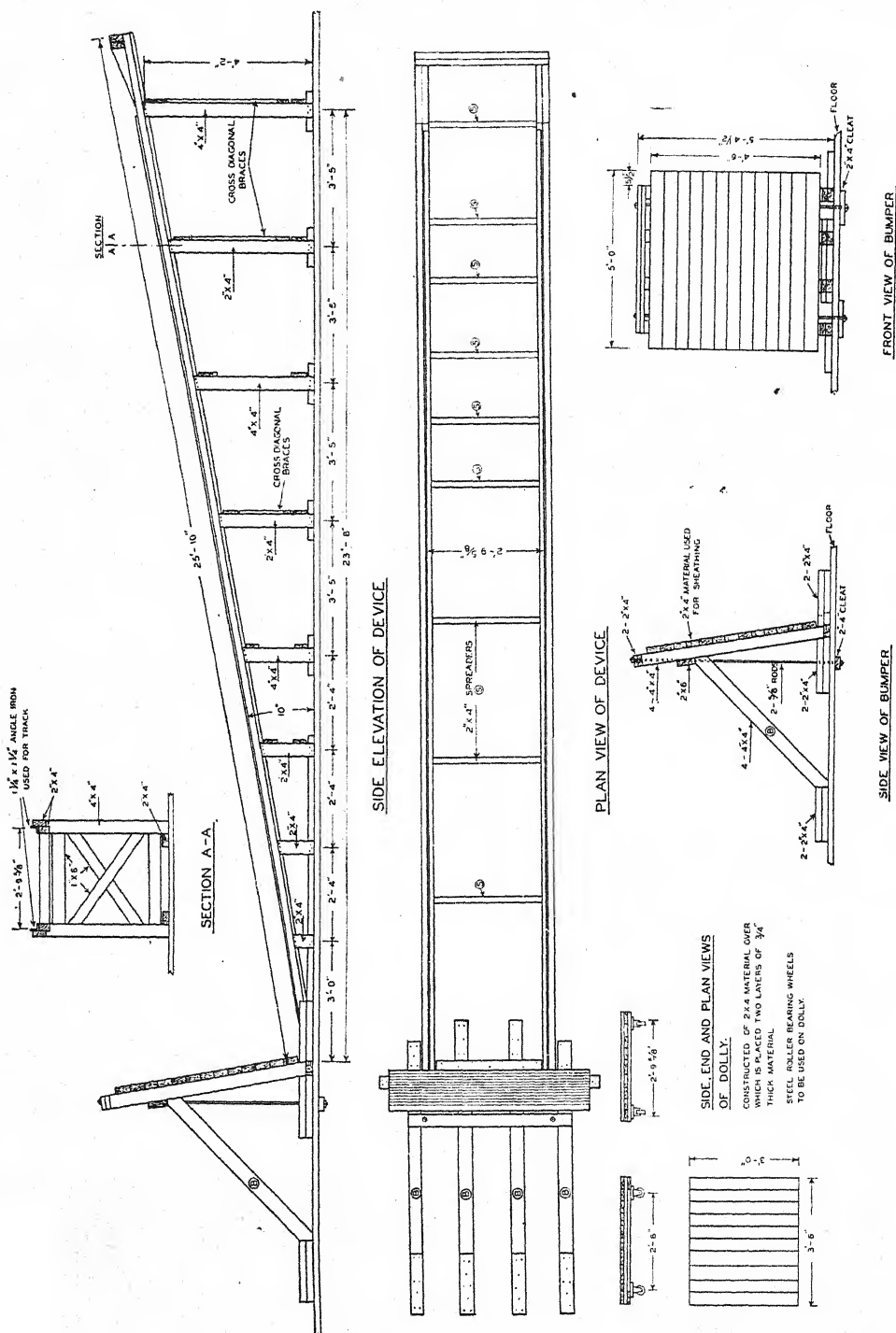
2. The apparatus shall consist of the following:

(a) *Inclined Track, Bumper, and Dolly*.—The principal apparatus consists of a two-rail steel track inclined 10 deg. from the horizontal, a rolling carriage or dolly, and a rigid bumper. The bumper shall be a wood barrier constructed at the bottom of the incline, with the plane of the face perpendicular to the direction of movement of the carriage. The bumper shall be equipped with a removable crosswise nominal 4 by 4-in. timber used as an optional hazard that can be so placed as to contact the container at the time of impact at any desired position between top and bottom edges of the container. The track shall accommodate the flat-bed rolling carriage or dolly which is equipped with steel wheels and a renewable wood or plywood face. (For tests of fiberboard containers the face of the dolly shall be covered with canvas.) The wood faces of the carriage and the bumper shall be maintained free of prominent projections which may affect the test results, such as bolt or nail

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-10 on Shipping Containers.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1946.





heads, scores, abrasions, and splits. The track shall be clean and the wheels well lubricated. The incline shall be graduated in increments of 6 in. and may be equipped with an automatic counting device which records each passage of the dolly to the lower end of the track. The apparatus may also have a cable and winch, or pulleys, to aid in bringing the dolly to the elevated end of the track and an automatic tripping device for releasing

### Calibration

3. (a) The impact test apparatus shall be fully calibrated so that the velocity attained at point of impact is known for each increment of height (or length of travel). A chart or graph shall be prepared from these values from which, for any desired velocity at impact, the height of the release point (or length of travel) may be selected.

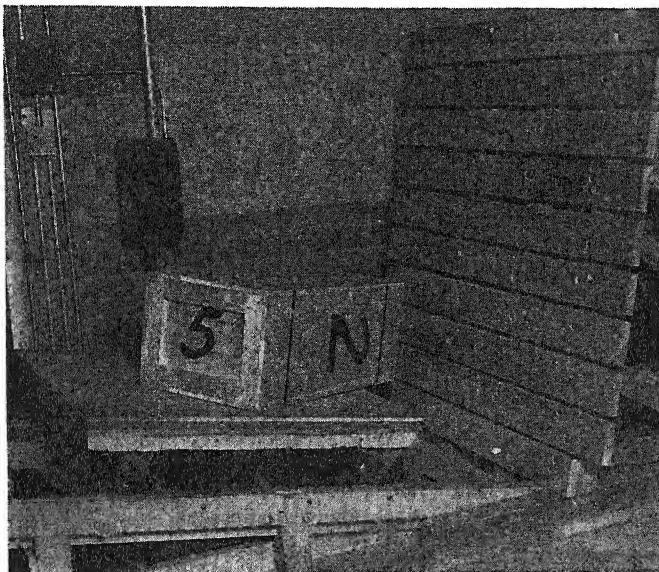


FIG. 2.—Impact on Edge of Container.

the dolly from a predetermined point on the incline. Construction details and a typical apparatus<sup>3</sup> are shown in Figs. 1, 2, and 3. The wheels shall be not less than 3 in. in diameter.

(b) *Conditioning Apparatus.*—Proper facilities for conditioning test containers at humidity and temperature in accordance with specification requirements.

(c) *Miscellaneous Equipment.*—Drying oven, scales, sharp knife, saw, etc., for taking samples for determination of the moisture content of the containers.

(b) The calibration shall be made on the empty dolly and may be made by any suitable apparatus such as an electric timing device by which the velocity may be determined in units of 0.01 ft. per sec. If a timing device is used, the average velocity shall be calculated from the known length of roll and the elapsed time. The velocity at impact shall be taken as twice the average velocity.

### Test Specimens

4. (a) When the container is being tested by Procedure A to determine the ability of the container to withstand

<sup>3</sup> A detailed description of this apparatus with construction drawings appears in *Freight Container Bulletin* 673, Freight Loading and Container Section, Assn. of Am. Railroads, 59 E. Van Buren St., Chicago 5, Ill.

impact stresses, it may be packed with either the actual contents or with a dummy load simulating such contents. When the container is being tested by Procedure B to determine the ability of the container or interior packing, or both, to provide protection to the contents, it shall be packed with the actual contents (Note) for which it was designed. In either procedure the container shall be

the far end as 6. The edges shall be identified by the numbers of the two faces which form that edge; for example, 1-2 identifies the edge where the top and right side meet, and 2-5 the edge formed by the right side and the near end. (In a fiberboard container, this edge will have the manufacturer's joint.) The corners shall be identified by the numbers of the three faces which meet to form that

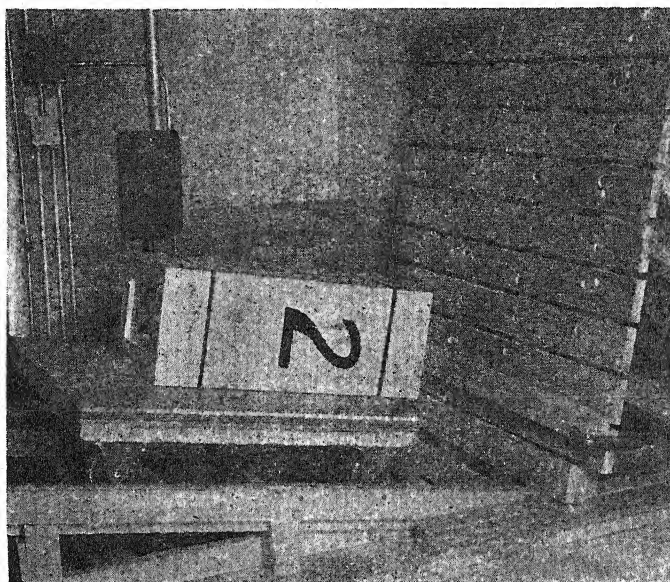


FIG. 3.—Impact on End of Container with 4 by 4-in. Timber in Position to Contact Lower Edge.

closed, strapped, or sealed in the same manner as will be used in actual shipment.

NOTE.—Certain contents or ingredients of the objects under test may be replaced by inert materials of approximately the same weight and characteristics.

(b) The faces, edges, and corners of the box shall be identified, as follows: Facing one end of the box (in the case of a fiberboard container, with the manufacturer's joint on the right), the top of the container shall be designated as 1, the right side as 2, the bottom as 3, the left side as 4, the near end as 5, and

corner; for example, 1-2-5 identifies the corner where the top, the right side, and the near end meet. This identification is illustrated in Fig. 4.

(c) In the case of other than rectangular containers, any convenient and adequate system of identification may be used.

#### Number of Tests

5. A minimum of five identical specimens, or more depending on the objectives of the test, shall be tested to obtain an average result.

### Conditioning

6. Depending on the purpose of the test, the containers may be conditioned prior to the incline impact test by either a different physical test, water immersion, exposure to water spray or exposure to fixed conditions of air temperature or

Fiberboard Containers (A.S.T.M. Designation: D 644).<sup>4</sup>

(b) *Wood*.—The moisture content of wood shall be determined by selecting a minimum of eight samples from different parts of the box, immediately following the test, of approximately 2 cu. in. each.

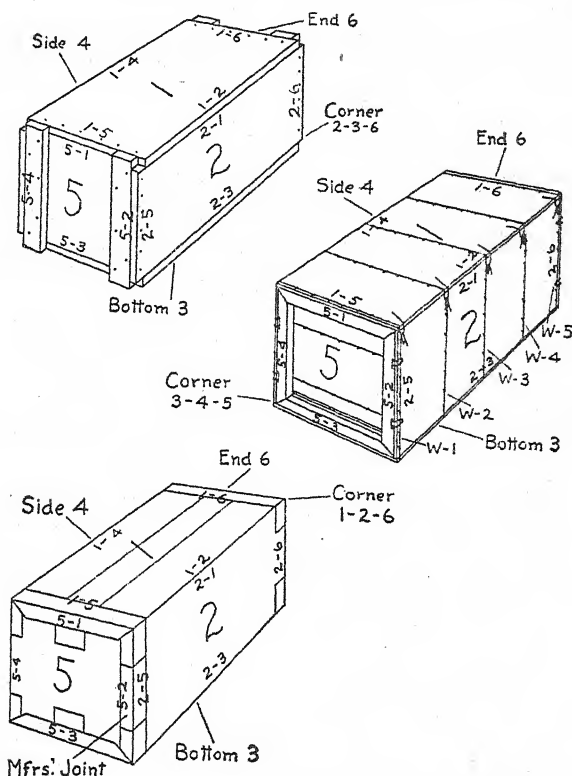


FIG. 4.—Identification of the Faces, Edges, and Corners of Test Specimen.

humidity, or other anticipated service conditions.

### Moisture Content

7. When the moisture content must be determined the following procedures shall be used:

(a) *Fiberboard*.—The moisture content of fiberboard at the time of test shall be determined in accordance with the Standard Method of Test for Moisture in Paper, Paperboard, and Paperboard and

They shall be weighed to the nearest 0.1 g., dried to constant weight at 100 to 105 C. (212 to 221 F.), and reweighed. The moisture content shall be expressed as a percentage of the oven-dry weight of the specimen.

### Distance of Travel

8. The distance from the bumper at which the dolly and container shall be released will depend upon the purpose

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book

of the test. The cycle of impacts may be started at any suitable distance, and succeeding cycles run from: (a) the same distance, or (b) from distances which are increased by equal increments for each cycle of impacts.

#### Procedure A

9. *To Determine the Ability of a Container to Withstand Impact Stresses.*—The container to be tested shall be placed on the dolly with the face or edge which is to receive the impact located either: (a) coincident with the forward end of the dolly or (b) projecting 2 in. beyond the forward end of the dolly. The dolly shall be brought to the predetermined position on the incline and released. If it is desired to concentrate the impact at any particular position on the container, the 4 by 4-in. timber shall be attached to the bumper in the desired position before the test. No part of the timber shall be struck by the dolly. The dolly and container shall then be drawn up the incline to the selected position and released. The position of the container on the dolly and the sequence in which the faces and edges are subjected to impacts may be at the option of the operator and will depend on the objective of the tests. The test shall be continued until failure of the container occurs. Depending on the purpose of the test, failure shall be considered to have occurred when:

- (1) The contents have spilled,
- (2) Any part of the contents may be removed from the container without damaging it further, or
- (3) Some predetermined type or types of damage have occurred to the container (Note).

NOTE.—It is presumed that if the contents are damaged, as determined by leakage of liquid contents or by other evidence, the test will be discontinued.

#### Procedure B

10. *To Determine the Ability of a Container or Interior Packing to Provide Protection to the Contents.*—This test shall be conducted as described in Section 9 until a predetermined number of impacts of prescribed intensity have been applied, whereupon the container shall be opened for inspection, unless previous evidence of failure is apparent.

#### Report

11. The report shall include the following:

(1) Dimensions of the container under test; its complete structural specifications, kind of wood, plywood or fiberboard, description and specifications for blocking and cushioning; spacing, size, and kind of fasteners; method of closing and strapping, if any; and the net and gross weights.

(2) Description of the contents of the container under test.

(3) A detailed record of test on each container, including damage to the container and contents, together with any other observation which may assist in correctly interpreting the results or aid in improving the design of the container or the method of packing, blocking, or cushioning.

(4) The method, if any, of conditioning the containers; the moisture content, if determined, of the wood, plywood, or fiberboard, and the results of any supplementary tests of the materials from which the container is made.

(5) A record of the calibration of the impact test apparatus.

(6) A statement to the effect that all tests were made in full compliance with this method, including a description of the location of specimens on the dolly, and an account as to the use of the optional hazard.

# *Tentative Method of Test for* SHIPPING CONTAINERS IN REVOLVING HEXAGONAL DRUM<sup>1</sup>



A.S.T.M. Designation: D 782 - 46 T

ISSUED, 1944; REVISED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. (a) This method<sup>3</sup> covers two procedures for performing tests on loaded shipping containers, as follows:

*Procedure A*, to determine the ability of the container to withstand rough handling, and

*Procedure B*, to determine the ability of the container to provide protection to its contents.

(b) This test is used to give an indication of the ability of a shipping container to withstand various shocks and impact stresses simulating those which may be expected in handling or shipment, or to protect its contents when subjected to such shocks and stresses, or to obtain average results permitting a comparison of different designs of containers of the same size and carrying the same load. The test permits an observation of the

progressive destruction or failure of the package, through which means of improving the design may be determined.

(c) This method is suitable for testing boxes, or crates made of metal, wood, fiberboard, or combinations of these materials.

## Apparatus

2. (a) The testing machine consists of a revolving drum which shall be in the form of a geometrical prism, as shown in Figs. 1 and 2 whose bases are regular hexagons and whose lateral faces are rectangles.<sup>4</sup> The axis of revolution shall be horizontal. Baffles or hazards shall be fixed on the inside faces of the drum. When prescribed in the specifications for the shipping container being tested, a conical projection shall be positioned on face 4 to simulate a puncture hazard. The inner faces of the drum shall be clean, smooth, and polished bright (Note). The drum shall be equipped with an automatic counting

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee D-10 on Shipping Containers.

<sup>2</sup> Revision accepted by the Administrative Committee on Standards, February 15, 1946.

<sup>3</sup> Further information on the development of this test appears in the paper by J. A. Newlin and T. R. C. Wilson, "The Development of a Box-Testing Machine and Some Results of Tests," *Proceedings, Am. Soc. Testing Mats.*, Vol. 16, Part II, p. 320 (1916).

<sup>4</sup> Prints of detailed drawings for the construction of the 7-ft. and 14-ft. diameter hexagonal drums shown in Figs. 1 and 2 are available at a nominal cost from the American Society for Testing Materials, 1916 Race St., Philadelphia 3, Pa.

device which will record six drops for each revolution.

NOTE—Wax is recommended to protect against rust when not in use.

(b) A drum 7 ft. in diameter (Fig. 1), revolving at a speed of  $1\frac{5}{8}$  rpm. about

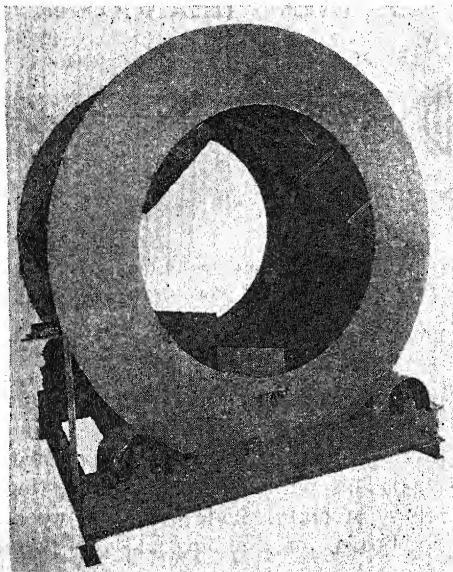


FIG. 1.—Box Testing Drum 7 ft. in Diameter.

its central axis, may be used for testing containers whose gross weight does not exceed 250 lb.

(c) A drum 14 ft. in diameter (Fig. 2), revolving at a speed of 1 rpm. about its central axis, may be used for testing containers whose weight does not exceed 600 lb.

### Test Specimens

3. (a) The container to be tested shall be packed with either (1) the actual contents for which it was designed, or (2) a dummy load simulating such contents. In either case the container shall be closed and sealed in the same manner as will be used in actual shipment.

(b) The faces, edges, and corners of the box shall be identified as follows: Facing one end of the box (in the case of a fiberboard container, with the manufacturer's joint on the right), the top of the container shall be designated as 1, the right side as 2, the bottom as 3, the

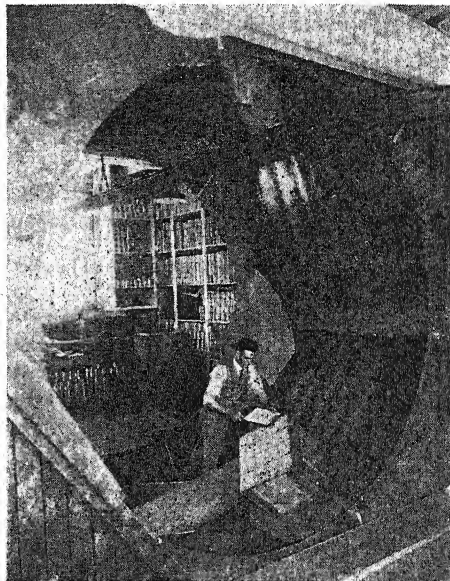


FIG. 2.—Box Testing Drum 14 ft. in Diameter.

left side as 4, the near end as 5, and the far end as 6. The edges shall be identified by the numbers of the two faces which form that edge; for example, 1-2 identifies the edge where the top and right side meet, and 2-5 the edge formed by the right side and the near end. (In a fiberboard container, this edge will have the manufacturer's joint.) The corners shall be identified by the numbers of the three faces which meet to form that corner; for example, 1-2-5 identifies the corner where the top, the right side, and the near end meet. This identification is illustrated in Fig. 3.

(c) Containers shall not be tested if their size and shape are such that they do



not move and fall freely when the drum is rotated.

### Number of Tests

4. A minimum of five but preferably ten identical specimens shall be tested to obtain an average result. The number of specimens tested may frequently be greater, depending on the objectives of the test.

containers shall be conditioned prior to testing in accordance with the Standard Method of Conditioning Paperboard, Fiberboard, and Paperboard Containers for Testing (A.S.T.M. Designation: D 641),<sup>5</sup> unless otherwise specified.

### Procedure A

6. *To Determine Ability of Container to Withstand Rough Handling.*—A record

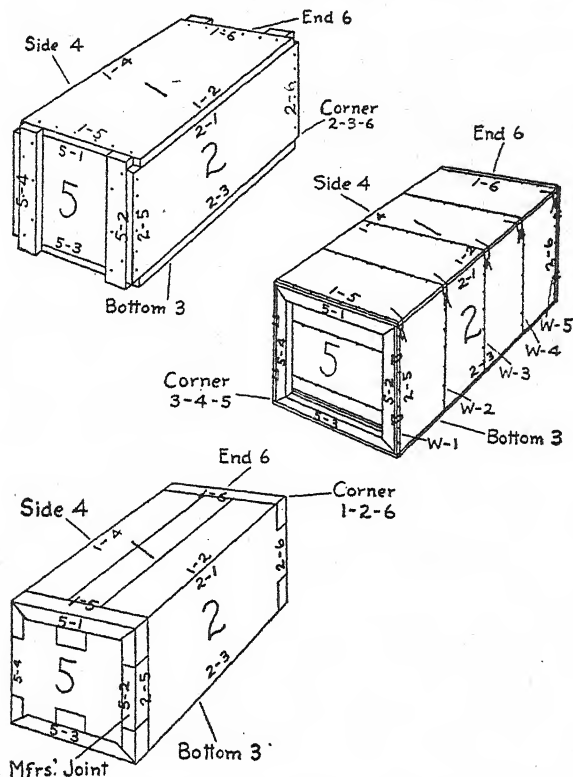


FIG. 3.—Identification of the Faces, Edges, and Corners of Test Specimen.

### Conditioning

5. Depending on the purpose of the test, the containers may be conditioned prior to the drum test by either a different physical test, water immersion, exposure to water spray, or exposure to fixed conditions of air temperature or humidity. For tests of permanent value and for comparisons between results obtained at various times and places,

shall be made of the preconditioning, if any, of the container and of its state at the beginning of the test. The test shall be started with the container placed in the crotch of the hazard on face No. 1 of the drum with the top face 1 of the container upward and with end 5 and side 2 against the sides of the hazard. The

<sup>5</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

drum shall be started in motion. As the drum revolves, the container slides, tumbles, and falls in varying positions from face to face of the drum, catching on and striking against the hazards and guides. As the test continues, a record shall be made of the development of damage to the container, such as skewing, puncture, splits or tears, and failure of the fastenings and reinforcements. This record shall be kept on a suitable data sheet. The drum may be stopped at intervals during the test to permit a close inspection of the container. Movement of the container from one face of the drum to the next shall be counted as one fall. The number of falls sustained by the container up to the time of occurrence of any damage shall be recorded with the description of the damage. In the record of the test, the faces, edges, and corners shall be designated as described in Section 3 (b). The test shall be continued until the container fails. Depending on the characteristics of the contents, failure shall be considered to have occurred, when:

- (1) The contents have spilled,
- (2) Any part of the contents may be removed from the container without damaging it further,
- (3) Some predetermined type or types of damage have occurred to the container, or
- (4) The contents have been damaged.

#### Procedure B

##### 7. To Determine Ability of Container

##### *to Provide Protection to Its Contents.—*

This test shall be conducted as described in Section 6, except that the drum may be stopped and the package opened for an inspection of the condition of the contents and interior blocking or packing material. Examinations may be made at the end of 6, 12, 24, or any other specified number of falls as desired. Record shall be made of the condition of the container and its contents.

#### Report

8. The report shall include the following:

- (1) Complete specifications of the containers tested, including type, style, material, inside and overall dimensions, and component parts, reinforcing, if any, number of fastenings or spacing,
- (2) Complete description of contents and inner packing materials including arrangement and fit,
- (3) Gross and net weights,
- (4) Detailed record of each test, including full observations of damage to container and contents and when it occurred,
- (5) Complete detail outline of test procedure, whether puncture hazard was used or not, and conditioning of containers prior to test, if any, and
- (6) All other observations which may assist in interpreting test results or in improving the design or construction of the container.

## *Tentative Specifications for*

### A.S.T.M. THERMOMETERS<sup>1</sup>



#### A.S.T.M. Designation: E 1 - 46 T

ISSUED, 1939; REVISED, 1940, 1941, 1942, 1943, 1944, 1945, 1946.<sup>2</sup>

These Tentative Specifications have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. These specifications cover glass thermometers graduated in Centigrade or Fahrenheit degrees and frequently specified in methods of the American Society for Testing Materials. The various thermometers covered are listed in Table I.

#### Requirements

2. The individual thermometers shall conform to the detailed specifications given in Table I and to the general requirements specified in Sections 3 to 11.

NOTE.—For the purpose of interpreting these specifications the following descriptions of terms apply:

The total length is the over-all length of the finished instrument.

The diameter is that measured with a ring gage or micrometer unless otherwise specified in Table I.

The length of the bulb is the distance from the bottom of the bulb to the beginning of the enamel backing.

<sup>1</sup> Under the standardization procedure of the Society, these specifications are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Latest revision accepted by the Administrative Committee on Standards, December 5, 1946.

These specifications when adopted as standard are intended to be incorporated in the Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1 - 46), see p. 454.

The top of the thermometer is the top of the finished instrument.

#### Type

3. Each thermometer shall be of the etched glass stem type unless otherwise specified in Table I.

#### Stem

4. The stem shall be made of suitable thermometer tubing and shall have a plain front and enamel back unless otherwise specified in Table I.

#### Bulb

5. The bulb shall be made of Corning normal or equally suitable thermometric glass.

#### Graduations

6. All graduation lines, figures, and letters shall be clear-cut and distinct. On partial immersion thermometers an immersion line of approximately the same length as the longest graduation line shall be etched on the front of the thermometer at the distance above the bottom of the bulb as specified in Table I.

#### Special Marking

7. The special markings on the thermometers in the form of an inscription as specified in Table I shall be in capital

letters without the use of periods. In addition to the special markings prescribed in Table I, each thermometer shall be marked with a serial number and the manufacturer's name or trade mark etched on the stem.

#### Scale Error

8. The error at any point of the scale up to the temperature limit prescribed in Table I when the thermometer is standardized in accordance with Section 9 shall not exceed the values prescribed in Table I.

#### Standardization

9. The thermometer shall be standardized immersed in the testing bath to

the top of the mercury column, unless other conditions of immersion are prescribed in Table I, and at the temperatures prescribed in Table I. (See Explanatory Notes.)

#### Test for Permanency of Range

10. The test for permanency of range shall be made at the temperature prescribed in Table I and under the immersion conditions specified for the thermometer. The accuracy after the test shall be within the limit specified.

#### Case

11. The thermometer shall be supplied in a suitable case on which shall appear the marking prescribed in Table I.

### EXPLANATORY NOTE ON PERIODIC CHECKING OF CHANGES OF BULB VOLUME OF GLASS THERMOMETERS

A mercury in glass thermometer, if properly made and used, is an extremely reliable instrument but small changes in volume of the thermometer bulb may occur, even though the thermometer may have been carefully annealed or aged.

Usually the bulb contracts slowly and as a result the thermometer may read higher after it has been placed in service than it did originally. These changes are more appreciable for short-range thermometers graduated in fractional degrees, and the users of such thermometers should check such instruments from time to time. When first received the thermometers should be checked every week or so, later these time intervals may be lengthened if the changes are not of consequence.

The ice-point method (reading in melting ice at 0 C. or 32 F.) has been found to be the most convenient and at the same time the most accurate method for checking changes in bulb volume. Other temperatures, such as the boiling point of water and of other pure substances, have been proposed for "fixed points," but have not been found entirely satisfactory owing to the difficulties of accurately obtaining these fixed temperatures.

Short-range thermometers graduated in fractional degrees may or may not be provided with an ice point reading if this temperature is not

included in the given range. This extra point can be incorporated in the thermometer, but may be omitted for the sake of economy. In the case of distillation thermometers intended to be inserted in the neck of a flask, the ice-point graduation may be undesirable since the contraction chamber between the ice point and the first graduation may contain enough mercury to cause uncertain and variable readings. For this reason the ice-point graduation, unless it is an integral part of the range, is generally omitted from the specifications for distillation thermometers.

In the absence of the ice point other means must be provided for detecting changes in the readings, especially if the thermometer is fractionally graduated. The method most commonly used involves the comparison at one or more temperatures in a well-stirred, properly constructed liquid bath of the thermometer to be checked against another standardized thermometer of similar specifications to the test thermometer but provided with an ice point.

Where several thermometers make up a series or set for some specific method and these thermometers do not have ice points, a similar set may be provided with ice points, and these should be kept for use as reference standards for checking routine test thermometers.

Detailed instructions for making such com-

parisons and specifications for suitable testing apparatus, as well as other data on this subject, will be found in the paper by E. F.

Mueller and R. M. Wilhelm, "Methods of Testing Thermometers," *Proceedings, Am. Soc. Testing Mats.*, Vol. 38, Part I, p. 493 (1938).

#### EXPLANATORY NOTE ON ICE POINT DETERMINATIONS OF GLASS THERMOMETERS

Two methods have been in use for determining the ice point on thermometers graduated fractionally and with an open scale, such as those of the Kinematic type. One method requires that the thermometer remain at approximately room temperature for 72 hr. before the ice point is taken. The other stipulates that the ice point reading be taken immediately or within a definitely stated short period after heating to the test temperature. Changes in the correction of the thermometer may be observed by determining the changes in the ice point by either method.

Since it may not be practical to wait until the thermometer has rested for three or more days, the method as outlined below is suggested.

These instructions are given in the form of a note which should appear in the table of corrections for the thermometer.

NOTE.—The tabulated corrections apply for the condition of immersion indicated provided the ice point reading taken after heating to ..... for not less than three minutes is ..... .

If the ice point reading (taken in not less than 2 min. and not more than 5 min. after removal of the thermometer from the heated bath) is found to be higher (or lower) than stated, all other readings will be higher (or lower) to the same extent.

(See Table I, pp. 1284 to 1291.)

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS.

Name.....	A.S.T.M. Kinematic Viscosity <sup>a</sup>	A.S.T.M. Kinematic Viscosity <sup>a</sup>	A.S.T.M. Kinematic Viscosity <sup>a</sup>
A.S.T.M. Thermometer.....	28F - 43T	29F - 43T	30F - 43T
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range..... For Tests at.....	97.5 to 102.5 F. 100 F.	127.5 to 132.5 F. 130 F.	207.5 to 212.5 F. 210 F.
Subdivisions.....	0.1 F.	0.1 F.	0.1 F.
Total Length.....	240 to 245 mm.	240 to 245 mm.	240 to 245 mm.
Stem Diameter.....	6.5 to 7.5 mm.	6.5 to 7.5 mm.	6.5 to 7.5 mm.
Bulb Diameter.....	6.0 mm. to not greater than stem	6.0 mm. to not greater than stem	6.0 mm. to not greater than stem
Bulb Length.....	45 to 55 mm.	45 to 55 mm.	45 to 55 mm.
Ice Point {	Range graduated.....	31.5 to 32.5 F.	31.5 to 32.5 F.
	Subdivisions.....	0.1 F.	0.1 F.
	Bottom of bulb to 32 F.....	77 to 87 mm.	77 to 87 mm.
Contraction Chamber {	Bottom of chamber to bottom of bulb, min....	100 mm.	100 mm.
	Top of chamber to bottom of bulb, max....	125 mm.	125 mm.
Expansion Chamber.....	permit heating to 220 F.	permit heating to 220 F.	permit heating to 220 F.
Bottom of Bulb to Graduation Line at..... Distance.....	97.5 F. 137 to 157 mm.	127.5 F. 137 to 157 mm.	207.5 F. 137 to 157 mm.
Top of Thermometer to Graduation Line at..... Distance.....	102.5 F. 27 to 47 mm.	132.5 F. 27 to 47 mm.	212.5 F. 27 to 47 mm.
Top Finish.....	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	0.5 F.	0.5 F.	0.5 F.
Graduations Numbered at Each Multiple of.....	1 F.	1 F.	1 F.
Immersion.....	total	total	total
Special Marking on Thermometer (See Section 7)	ASTM KIN VIS	ASTM KIN VIS	ASTM KIN VIS
Scale Error at..... when standardized, shall not exceed.....	100 F. 0.10 F.	130 F. 0.10 F.	210 F. 0.10 F.
Standardization.....	The thermometers shall be standardized at the ice point, <sup>b</sup> and at the viscosity test point (100, 130, or 210 F.) for conditions of total immersion, expressed to the nearest 0.02 F. and such corrections shall be added to the observed reading.		
Marking on Case.....	A.S.T.M. Kinematic Viscosity Thermometer 97.5 to 102.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 127.5 to 132.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 207.5 to 212.5 F.

<sup>a</sup> Thermometers conforming to the requirements for A.S.T.M. Kinematic Viscosity Thermometers E 1 (28F-39T), E 1 (29F-39T), and E 1 (30F-39T) formerly prescribed in the Tentative Specifications for A.S.T.M. Thermometers (A S T M. Designation: E 1 - 42 T), 1942 Book of A.S.T.M. Standards, Part II, p. 1408, Part III, p. 1555, shall be considered as meeting these specifications.

<sup>b</sup> See Explanatory Note on Ice Point Determinations of Glass Thermometers which appears in the text.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Aniline Point <sup>a</sup>	A.S.T.M. Aniline Point <sup>a</sup>	A.S.T.M. Aniline Point <sup>a</sup>
A.S.T.M. Thermometer.....	33C - 41T	34C - 41T	35C - 41 T
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-38 to +42 C.	25 to 105 C.	90 to 170 C.
Subdivisions.....	0.2 C.	0.2 C.	0.2 C.
Total Length.....	403 to 409 mm.	403 to 409 mm.	403 to 409 mm.
Stem Diameter.....	6.0 to 7.0 mm.	6.0 to 7.0 mm.	6.0 to 7.0 mm.
Bulb Diameter.....	5.0 to 6.0 mm.	5.0 to 6.0 mm.	5.0 to 6.0 mm.
Bulb Length.....	10 to 20 mm.	10 to 20 mm.	10 to 20 mm.
Bottom of Bulb to Graduation Line at.....	-38 C.	25 C.	90 C.
Distance.....	101 to 115 mm.	101 to 115 mm.	101 to 115 mm.
Top of Thermometer to Graduation Line at.....	42 C.	105 C.	170 C.
Distance.....	25 to 45 mm.	25 to 45 mm.	25 to 45 mm.
Contraction Chamber.....	....	b	b
Expansion Chamber.....	yes	yes	yes
Top Finish.....	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	1 C.	1 C.	1 C.
Graduations Numbered at Each Multiple of.....	2 C.	2 C.	2 C.
Immersion (See Section 6).....	51 mm.	51 mm.	51 mm.
Special Marking on Thermometer (See Section 7).....	ASTM ANILINE POINT 51 MM IMM	ASTM ANILINE POINT 51 MM IMM	ASTM ANILINE POINT 51 MM IMM
Scale Error at any point when standardized shall not exceed..	0.2 C.	0.2 C.	0.2 C.
Standardization.....	c	c	c
Marking on Case.....	A.S.T.M. Aniline Point Thermometer -38 to +42 C.	A.S.T.M. Aniline Point Thermometer 25 to 105 C.	A.S.T.M. Aniline Point Thermometer 90 to 170 C.

<sup>a</sup> Editorially revised in 1942.<sup>b</sup> The top of the contraction chamber shall be not more than 35 mm. above the bottom of the bulb. The mercury should stand at approximately the center of the contraction chamber at 0 C.<sup>c</sup> The aniline point thermometers shall be standardized for 51-mm. immersion and for the following temperatures of the emergent mercury column:

- 38 to + 42 C. Thermometer		25 to 105 C. Thermometer		90 to 170 C. Thermometer	
Standardization Temperature	Average Temperature of Emergent Mercury Column	Standardization Temperature	Average Temperature of Emergent Mercury Column	Standardization Temperature	Average Temperature of Emergent Mercury Column
-35 C.....	5 C.				
-20 C.....	15 C.	25 C.....	25 C.	100 C.....	70 C.
0 C.....	20 C.	50 C.....	40 C.	130 C.....	60 C.
20 C.....	25 C.	75 C.....	45 C.	160 C.....	50 C.
40 C.....	30 C.	100 C.....	45 C.		



TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation
A.S.T.M. Thermometer.....	37C - 44T	38C - 44T	39C - 44T
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	-2 to +52 C.	24 to 78 C.	48 to 102 C.
Subdivisions.....	0.2 C.	0.2 C.	0.2 C.
Total Length.....	380 to 385 mm.	380 to 385 mm.	380 to 385 mm.
Stem Diameter.....	6.0 to 7.0 mm.	6.0 to 7.0 mm.	6.0 to 7.0 mm.
Bulb Diameter.....	not greater than stem	not greater than stem	not greater than stem
Bulb Length.....	15 to 20 mm.	15 to 20 mm.	15 to 20 mm.
Bottom of Bulb to Graduation Line at Distance.....	-2 C. 125 to 145 mm.	24 C. 125 to 145 mm.	48 C. 125 to 145 mm.
Top of Thermometer to Graduation Line at Distance.....	52 C. 25 to 45 mm.	78 C. 25 to 45 mm.	102 C. 25 to 45 mm.
Contraction Chamber.....	none	a	a
Expansion Chamber.....	b	b	b
Top Finish.....	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	1 C.	1 C.	1 C.
Graduations Numbered at Each Multiple of.....	2 C.	2 C.	2 C.
Immersion (See Section 6).....	100 mm.	100 mm.	100 mm.
Special Marking on Thermometer (see Section 7).....	ASTM SOLVENTS DIST 100 MM IMM	ASTM SOLVENTS DIST 100 MM IMM	ASTM SOLVENTS DIST 100 MM IMM
Scale Error at any point when standardized shall not exceed.....	0.2 C.	0.2 C	0.2 C.
Standardization.....	c, d	c, d	c, d
Marking on Case.....	A.S.T.M. Solvents Distillation Thermometer, -2 to +52 C., 100-mm. immersion	A.S.T.M. Solvents Distillation Thermometer, 24 to 78 C., 100-mm. immersion	A.S.T.M. Solvents Distillation Thermometer, 48 to 102 C., 100-mm. immersion

<sup>a</sup> The top of the contraction chamber shall be not more than 35 mm. above the bottom of the bulb. The mercury shall stand near the bottom of the contraction chamber at 0 C.

<sup>b</sup> The expansion chamber shall permit heating the thermometer at least 25 C. above the highest temperature on the scale.

<sup>c</sup> For precautions to be observed in determining changes in bulb volume see Explanatory Note on Periodic Checking of Changes of Bulb Volume of Glass Thermometers, which appears in the text.

<sup>d</sup> The A.S.T.M. Solvents Distillation Thermometers shall be standardized for 100-mm. immersion at the temperatures specified below and for the following average temperatures of the emergent mercury column:

-2 to +52 C. Thermometer		24 to 78 C. Thermometer		48 to 102 C. Thermometer	
Standardization Temperature	Average Temperature of Emergent Mercury Column	Standardization Temperature	Average Temperature of Emergent Mercury Column	Standardization Temperature	Average Temperature of Emergent Mercury Column
25 C.....	25 C.	25 C.....	25 C.	50 C.....	30 C.
50 C.....	25 C.	50 C.....	25 C.	75 C.....	30 C.
		75 C.....	25 C.	100 C.....	30 C.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation	A.S.T.M. Solvents Distillation
A.S.T.M. Thermometer.....	40C - 44T	41C - 44T	42C - 44T
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	72 to 126 C.	98 to 152 C.	95 to 255 C.
Subdivisions.....	0.2 C.	0.2 C.	0.5 C.
Total Length.....	380 to 383 mm.	380 to 385 mm.	380 to 385 mm.
Stem Diameter.....	6.0 to 7.0 mm.	6.0 to 7.0 mm.	6.0 to 7.0 mm.
Bulb Diameter.....	not greater than stem	not greater than stem	not greater than stem
Bulb Length.....	15 to 20 mm.	15 to 20 mm.	15 to 20 mm.
Bottom of bulb to Graduation Line at..... Distance.....	72 C. 125 to 145 mm.	98 C. 125 to 145 mm.	95 C. 125 to 145 mm.
Top of Thermometer to Graduation Line at..... Distance.....	126 C. 25 to 45 mm.	152 C. 25 to 45 mm.	255 C. 25 to 45 mm.
Contraction Chamber.....	a	a	a
Expansion Chamber.....	b	b	b
Top Finish.....	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	1 C.	1 C.	1 C.
Graduations Numbered at Each Multi- ple of.....	2 C.	2 C.	5 C.
Immersion (See Section 6).....	100 mm.	100 mm.	100 mm.
Special Marking on Thermometer (See Section 7).....	ASTM SOLVENTS DIST 100 MM IMM	ASTM SOLVENTS DIST 100 MM IMM	ASTM SOLVENTS DIST 100 MM MM
Scale Error at any point when standard- ized shall not exceed.....	0.2 C.	0.2 C.	0.5 C.
Standardization.....	c, d	c, d	c, d
Marking on Case.....	A.S.T.M. Solvents Dis- tillation Thermometer, 72 to 126 C., 100-mm. immersion	A.S.T.M. Solvents Dis- tillation Thermometer, 98 to 152 C., 100-mm. immersion	A.S.T.M. Solvents Dis- tillation Thermometer, 95 to 255 C., 100-mm. immersion

<sup>a</sup> The top of the contraction chamber shall be not more than 35 mm. above the bottom of the bulb. The mercury shall stand near the bottom of the contraction chamber at 0 C.

<sup>b</sup> The expansion chamber shall permit heating the thermometer at least 25 C. above the highest temperature on the scale.

<sup>c</sup> For precautions to be observed in determining changes in bulb volume see Explanatory Note on Periodic Checking of Changes of Bulb Volume of Glass Thermometers, which appears in the text.

<sup>d</sup> The A.S.T.M. Solvents Distillation Thermometers shall be standardized for 100-mm. immersion at the temperatures specified below and for the following average temperatures of the emergent mercury column:

72 to 126 C. Thermometer		98 to 152 C. Thermometer		95 to 255 C. Thermometer	
Standardization Temperature	Average Tempera- ture of Emergent Mercury Column	Standardization Temperature	Average Tempera- ture of Emergent Mercury Column	Standardization Temperature	Average Tempera- ture of Emergent Mercury Column
75 C.....	30 C.	100 C.....	30 C.	100 C.....	30 C.
100 C.....	30 C.	125 C.....	35 C.	150 C.....	35 C.
125 C.....	30 C.	150 C.....	35 C.	200 C.....	40 C.
				250 C.....	45 C.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Kinematic Viscosity	A.S.T.M. Kinematic Viscosity	A.S.T.M. Kinematic Viscosity
A.S.T.M. Thermometer.....	43 F - 45 T	44 F - 46 T	45 F - 46 T
Liquid.....	mercury-thallium	mercury	mercury
Filling above Liquid.....	nitrogen or other suitable gas	nitrogen gas	nitrogen gas
Temperature Range..... For Tests at.....	-61 to -29 F. ....	66.5 to 71.5 F. 68 F. and 70 F.	74.5 to 79.5 F. 77 F.
Subdivisions.....	0.2 F.	0.1 F.	0.1 F.
Total Length.....	400 to 412 mm.	240 to 245 mm.	240 to 245 mm.
Stem Diameter.....	7.0 to 8.0 mm.	6.5 to 7.5 mm.	6.5 to 7.5 mm.
Bulb Diameter.....	6.0 to 7.0 mm.	6.0 mm. to not greater than stem	6.0 mm. to not greater than stem
Bulb Length.....	30 to 40 mm.	45 to 55 mm.	45 to 55 mm.
Ice Point {	Range graduated.....	+31 to +33 F.	31.5 to 32.5 F.
	Subdivisions.....	0.2 F.	0.1 F.
	Bottom of bulb to +32 F. ....	320 to 375 mm.	77 to 87 mm.
Contraction Chamber {	Bottom of chamber to bottom of bulb, min.....	290 mm. <sup>a</sup>	100 mm.
	Top of chamber to bottom of bulb, max.....	310 mm. <sup>a</sup>	125 mm.
Expansion Chamber.....	<sup>b</sup>	permit heating to 220 F.	permit heating to 220 F.
Bottom of Bulb to Graduation Line at.... Distance.....	-61 F. 60 to 90 mm.	66.5 F. 137 to 157 mm.	74.5 F. 137 to 157 mm.
Top of Thermometer to Graduation Line at. Distance.....	-29 F. 126 to 156 mm.	71.5 F. 27 to 47 mm.	79.5 F. 27 to 47 mm.
Top Finish.....	plain	glass ring	glass ring
Longer Graduation Lines at Each.....	1 F.	0.5 F.	0.5 F.
Graduations Numbered at Each Multiple of	2 F. starting at -60 F.	1 F.	1 F.
Immersion.....	total	total	total
Special Marking on Thermometer.....	ASTM KIN VIS	ASTM KIN VIS	ASTM KIN VIS
Scale Error at..... when standardized, shall not exceed.....	any point 0.2 F.	68 and 70 F. 0.10 F.	77 F. 0.10 F.
Standardization.....	ice point <sup>c</sup> and at every 10 F. of the calibrated interval starting at -60 F.	The thermometers shall be standardized at the ice point, <sup>c</sup> and at the viscosity test point (68 and 70, or 77 F.) for conditions of total immersion, expressed to the nearest 0.02 F. and such corrections shall be added to the observed reading.	
Marking on Case.....	A.S.T.M. Kinematic Viscosity Thermometer -61 to -29 F.	A.S.T.M. Kinematic Viscosity Thermometer 66.5 to 71.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 74.5 to 79.5 F.

<sup>a</sup> The length of unchanged capillary between the nearest graduation mark and the contraction chamber shall be not less than 10 mm.

<sup>b</sup> The expansion chamber shall be of the long, narrow type 10 to 20 mm. in length. The length of unchanged capillary between the nearest graduation mark and the expansion chamber shall be not less than 10 mm. The expansion chamber shall permit heating the thermometer to 220 F.

<sup>c</sup> See Explanatory Note on Ice Point Determinations of Glass Thermometers which appears in the text.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Kinematic Viscosity	A.S.T.M. Kinematic Viscosity	A.S.T.M. Kinematic Viscosity
A.S.T.M. Thermometer.....	46 F - 46 T	47 F - 46 T	48 F - 46 T
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range..... For Tests at.....	119.5 to 124.5 F. 122 F.	137.5 to 142.5 F. 140 F.	177.5 to 182.5 F. 180 F.
Subdivisions.....	0.1 F.	0.1 F.	0.1 F.
Total Length.....	240 to 245 mm.	240 to 245 mm.	240 to 245 mm.
Stem Diameter.....	6.5 to 7.5 mm.	6.5 to 7.5 mm.	6.5 to 7.5 mm.
Bulb Diameter.....	6.0 mm. to not greater than stem	6.0 mm. to not greater than stem	6.0 mm. to not greater than stem
Bulb Length.....	45 to 55 mm.	45 to 55 mm.	45 to 55 mm.
Ice Point { Range graduated..... Subdivisions..... Bottom of bulb to 32 F. (0 C.)..	31.5 to 32.5 F. 0.1 F. 77 to 87 mm.	31.5 to 32.5 F. 0.1 F. 77 to 87 mm.	31.5 to 32.5 F. 0.1 F. 77 to 87 mm.
Contraction Chamber { Bottom of chamber to bottom of bulb, min..... Top of chamber to bottom of bulb, max.....	100 mm. 125 mm.	100 mm. 125 mm.	100 mm. 125 mm.
Expansion Chamber.....	permit heating to 220 F.	permit heating to 220 F.	permit heating to 220 F.
Bottom of Bulb to Graduation Line at..... Distance.....	119.5 F. 137 to 157 mm.	137.5 F. 137 to 157 mm.	177.5 F. 137 to 157 mm.
Top of Thermometer to Graduation Line at..... Distance.....	124.5 F. 27 to 47 mm.	142.5 F. 27 to 47 mm.	182.5 F. 27 to 47 mm.
Top Finish.....	glass ring	glass ring	glass ring
Longer Graduation Lines at Each.....	0.5 F.	0.5 F.	0.5 F.
Graduations Numbered at Each Multiple of.....	1 F.	1 F.	1 F.
Immersion.....	total	total	total
Special Marking on Thermometer.....	ASTM KIN VIS	ASTM KIN VIS	ASTM KIN VIS
Scale Error at..... when standardized, shall not exceed.....	122 F. 0.10 F.	140 F. 0.10 F.	180 F. 0.10 F.
Standardization.....	The thermometers shall be standardized at the ice point,* and at the viscosity test point (122, 140, or 180 F.) for conditions of total immersion, expressed to the nearest 0.02 F. and such corrections shall be added to the observed reading.		
Marking on Case.....	A.S.T.M. Kinematic Viscosity Thermometer 119.5 to 124.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 137.5 to 142.5 F.	A.S.T.M. Kinematic Viscosity Thermometer 177.5 to 182.5 F.

\* See Explanatory Note on Ice Point Determinations of Glass Thermometers which appears in the text.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS (Continued).

Name.....	A.S.T.M. Gas Calorimeter Inlet	A.S.T.M. Gas Calorimeter Outlet	A.S.T.M. Gas Calorimeter Flue
A.S.T.M. Thermometer.....	50 F - 46 T	51 F - 46 T	52 F - 46 T
Liquid.....	mercury	mercury	mercury
Filling above Liquid.....	nitrogen gas	nitrogen gas	nitrogen gas
Temperature Range.....	54 to 101 F.	69 to 116 F.	54 to 101 F.
Subdivisions.....	0.1 F.	0.1 F.	1 F.
Total Length.....	457 to 463 mm.	457 to 463 mm.	205 to 210 mm.
Stem Diameter.....	6.0 to 7.0 mm.	6.0 to 7.0 mm.	6.0 to 7.0 mm.
Bulb Diameter.....	7.0 to 8.0 mm. (cylindrical shape)	7.0 to 8.0 mm. (cylindrical shape)	not greater than stem
Bulb Length.....	20 to 25 mm.	20 to 25 mm.	20 to 25 mm.
Bottom of Bulb to Graduation Line at..... Distance.....	54 F. 100 to 120 mm.	69 F. 100 to 120 mm.	54 F. 90 to 105 mm.
Top of Thermometer to Graduation Line at..... Distance.....	101 F. 25 to 45 mm.	116 F. 25 to 45 mm.	101 F. 20 to 35 mm.
Top Finish.....	plain	plain	plain
Bottom of Bulb to Top of Contraction Chamber.....	not over 36 mm. mercury to stand in chamber at 32 F.	not over 36 mm. mercury to stand in chamber at 32 F.	none
Expansion Chamber.....	permit heating to 150 F.	permit heating to 212 F.	permit heating to 150 F.
Longer Graduation Lines at Each.....	0.5 F.	0.5 F.	5 F.
Graduations Numbered at Each Multiple of.....	1 F.	1 F.	10 F.
Immersion.....	total	total	total
Special Marking on Thermometer.....	ASTM GAS CAL INLET	ASTM GAS CAL OUTLET	ASTM GAS CAL FLUE
Scale Error at any point when standardized, shall not exceed.....	0.2 F.	0.2 F.	0.5 F.
Change in Correction over any 0.5 F. Interval shall not exceed.....	0.10 F.	0.10 F.	....
Standardization.....	at multiples of 5 F.	at multiples of 5 F.	at 60 and 90 F.
Marking on Case.....	A.S.T.M. Gas Calorimeter Inlet Thermometer 54 to 101 F.	A.S.T.M. Gas Calorimeter Outlet Thermometer 69 to 116 F.	A.S.T.M. Gas Calorimeter Flue Thermometer 54 to 101 F.

TABLE I.—REQUIREMENTS FOR A.S.T.M. THERMOMETERS  
(Concluded).

Name.....	A.S.T.M. Congealing Point
A.S.T.M. Thermometer.....	54 F - 46 T
Liquid.....	mercury
Filling above Liquid.....	nitrogen gas
Temperature Range.....	68 to 212 F.
Subdivisions.....	0.5 F. <sup>a</sup>
Total Length.....	295 to 305 mm.
Stem Diameter.....	6.0 to 7.0 mm.
Bulb Shape.....	Ellipsoidal (see Fig. 1)
Bulb Diameter.....	4.5 to 6.0 mm.
Bulb Length.....	10 to 12 mm.
Expansion Chamber.....	permit heating to 230 F.
Bottom of Bulb to Graduation Line at.....	68 F.
Distance.....	55 to 75 mm.
Top of Thermometer to Graduation Line at.....	212 F.
Distance.....	30 to 50 mm.
Top Finish.....	glass ring
Longer Graduation Lines at Each.....	1 F.
Graduations Numbered at Each Multiple of.....	5 F.
Immersion.....	total
Special Marking on Thermometer.....	ASTM CONGEALING PT
Scale Error at any point when standardized shall not exceed.....	0.5 F.
Standardization.....	70, 140, and 210 F.
Marking on Case.....	A.S.T.M. Congealing Point Thermometer 68 to 212 F.

<sup>a</sup> For purposes of calibration and certification two or three division markings shall be placed above the 212 F. mark.

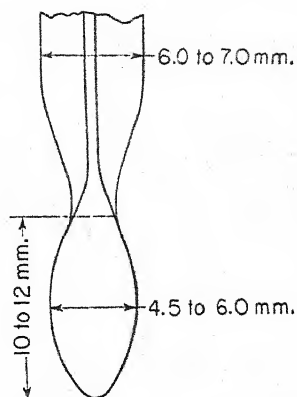


FIG. 1.—Bulb of A.S.T.M. Congealing Point Thermometer.

# *Tentative Method of Test for* PARTICLE SIZE DISTRIBUTION OF SUBSIEVE SIZE PARTICULATE SUBSTANCES<sup>1</sup>



A.S.T.M. Designation: E 20 - 33 T

ISSUED, 1933.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

## Scope

1. This method of test covers the procedure for determining the particle size of particulate substances in absolute units, as far as the particle shape will permit. It is intended to cover the range of sizes between the 74-micron (No. 200) sieve and 0.2 micron. The method is applicable in its entirety to homogeneous materials. In the case of mixtures, the extent of application is limited by the properties of the components of the mixture.

## Definitions

2. (a) *Dispersion*.—In microscopy it refers to the distribution of the individual particles. In statistical work it is measured by the standard deviation.

NOTE.—Both uses of the word seem too generally accepted to warrant a change in either.

(b) *Individual Particle*.—Those minute units of matter (of which the material is composed) whose size and shape

depend only on the force of cohesion. Such particles cannot be subdivided without separating like molecules that are within the range of the cohesive attraction of one another.<sup>3</sup>

(c) *Aggregate*.—A group of two or more attached particles.

NOTE.—There are three forms of aggregates: namely, ultimate working unit, flocculate, and apparent flocculate.

(d) *Ultimate Working Unit*.—An individual particle or any group of individual particles that are so firmly held together by forces of adhesion that they remain intact as a group throughout the duration of their application.

(e) *Flocculate*.—Flocculation occurs only where particles have been incorporated in a liquid or plastic medium. A flocculate is a group of particles held together by a force apparently arising from interfacial tension. This force of flocculation is relatively weak, consequently flocculates do not function as large individual particles, and cannot be classified under ultimate working units.

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1933. Editorially revised and rearranged in 1939.

<sup>3</sup> Henry Green, *Journal of Industrial Hygiene*, Vol. 7, p. 155 (1925).



Under slight, continuously applied force flocculates are constantly breaking down and reforming.

*Apparent Flocculates* as found in gas-floated or air-floated particles show a somewhat similar type of aggregation. Usually the force of attraction between the particles is relatively weak and the particles can readily be dispersed in suitable media. For practical purposes, the more inclusive term, aggregate, is preferable to flocculate for gas-floated or air-floated particles.

(f) *Average Diameter*.—The diameter of a hypothetical particle which, in some particular way, represents the total mass of particles.

(g) *Nonuniformity*.—A nonuniform particulate substance is one in which the constituent particles differ from one another in diameters.

#### Rough Separation of Sample into Size Groups

3. (a) For pigments or other materials which are essentially less than 6 microns, separation of the sample is not necessary and the mount shall be prepared in accordance with Section 4.

(b) For wide ranges of distribution of particle size in subsieve size portions, auxiliary separation into groups of sizes is necessary to facilitate measurement with the microscope. Rough separation is permissible as exact size measurement in microns follows. The following general ranges of particle diameter are proposed for the separation:

A.....	24 microns and larger
B.....	24 to 12 microns
C.....	12 to 6 microns
D.....	6 to 0.2 microns

For specific purposes A or D may be further separated.

NOTE.—If the sample contains particles larger than the 74-micron (No. 200) sieve, the

material shall be separated by sieving wet in a suitable fluid. The portion which passes the sieve may be recovered by evaporation of the fluid. The portion which remains on the sieve may be subjected when dry to a standard sieve test and additional material that passes the 74-micron (No. 200) sieve added to that which is to be tested.

(c) Separation of the sample shall be made by air or liquid elutriation or by sedimentation at room temperature. The volume of liquid in the elutriator or sedimentation apparatus should be between 20 and 100 ml. per gram of solid. With finer particles and with material of lesser density the greater volumes should be used. The technique involved in the elutriation or settling is necessarily influenced by the type of material being measured and the dispersing liquids. The size of the vessel and dispersion will be specified in terms of the uniformity obtained. The microscope measurement will be used as a check upon the effectiveness of the separation. For accurate results at least 50 per cent by weight must fall within the boundaries of the suggested ranges set for the elutriation. Of the material falling outside the boundaries, not over 5 per cent by weight may fall outside of a range which is one half of the lower limit and twice the upper limit of the range under consideration.

(d) The fluid used in the elutriation or sedimentation shall contain no dissolved solids which would influence the weight determination of a given portion, that is, the fluid shall be 100 per cent volatile. It shall not react chemically with the material being elutriated. Water, kerosine, alcohols, and the like may be used. It is suggested that the filtering of any elutriator portion should be through alundum to avoid contamination of the sample with paper or pulp fibers. As an alternative, the solid may be recovered from each stage of the elutriator by evaporation of a concentrated slurry,

provided the temperatures used for the evaporation do not alter the specific product. The total weight of the portions recovered shall check within 5 per cent the weight of material initially placed in the elutriator.

(e) All limits are specified to insure good microscopic technique. Good elutriation technique for analytical purposes falls well within these limits.

NOTE.—Quite obviously, materials containing components of different densities will elutriate or settle in ranges of low uniformity. The present requirements can only be applied when the material meets the requirements for homogeneity. If the materials can be readily identified under the microscope, they may be measured individually and the results considered as exact, notwithstanding wide differences in density between two materials which may cause lack of uniformity. In other cases, such as cement or coal, where the presence respectively of gypsum and ash constituents may affect the test, these effects may be considered negligible for the normal type of products.

#### Preparation of Mount

4. Any one of a number of methods of preparing the mount may be used, the criterion of suitability being conformity to the following requirements:

The particles shall be essentially in one plane.

The particles shall be free from motion.

The particles shall be dispersed, showing individual grains instead of aggregates and flocculates.

The particles shall not be ground in mounting.

The mount shall be truly representative of the distribution of sizes in the material.

The mounting medium shall be selected to give maximum definition.

(a) *Fine Materials, Pigments, etc.*—The type of mount that should be used depends on the physical properties of the particles, and for this reason no definite

requirements are specified. The general procedure shall be to place the material being tested on a microscope slide, and then to rub it out in a solution of gum or resin in a solvent until the particles are well dispersed. After the solvent has evaporated, the nonvolatile constituent of the dispersing agent serves to cement the particles to the slide in the dispersed state.

NOTE.—Detailed technique will be found in the work of Dunn<sup>4</sup> and Green.<sup>5</sup> The former uses balsam in xylol, and the latter turpentine and dammar. Other mounting media suggested are 0.5 per cent glycerol in alcohol, rubber cement, styra<sup>x</sup> in xylol, and the chlorinated naphthalenes and saponin.

(b) *Coarse Material, Ground Products, etc.*—In the case of coarse materials it is often advantageous to use the so-called dry mount, in which the dispersing agent used is volatile and can be driven off after the material has been dispersed on the slide. The uniformity of the mount when examined microscopically is the best criterion of an acceptable mount.

#### Procedure

5. (a) *Direct Observation Method.*—Direct observations using the Filar micrometer<sup>6</sup> has been used with fair success and reasonable degree of accuracy on the coarser fraction of particulate substances. The Filar micrometer is an attachment which fits into the draw tube of the microscope and is fitted with a Ramsden-type eyepiece which permits focusing on a movable cross hair activated by a micrometer screw. This instrument permits direct measurement of the particle as viewed on the slide. This method, however, is

<sup>4</sup> E. J. Dunn, Jr., *Industrial and Engineering Chemistry*, Analytical Edition, Vol. 2, p. 59 (1930).

<sup>5</sup> Henry Green, *Industrial and Engineering Chemistry*, Vol. 16, No. 7, p. 677 (1924).

<sup>6</sup> J. C. Pearson and W. H. Sligh, National Bureau of Standards *Technological Paper No. 48* (1915).

not recommended for materials in the 0.2 to 6 micron class size.

(b) *Projection Method*.—The projection method<sup>4</sup> which involves throwing the image of the particle on a screen has several distinct advantages. It further magnifies the image and permits focusing through the depth of the mount, insuring a proper focus and count of all the particles.

(c) *Photomicrographic Method*.—The photomicrographic method,<sup>7,8</sup> which involves taking a photomicrograph of the particles and subsequently measuring them on a print or by projecting the negative on the screen, is also recommended. This method has the very decided advantage of making a permanent record of the sample and permits eye comparisons of two or more samples. In the use of this method it is extremely important that the mount be made according to the procedure outlined under the method of Green<sup>7</sup> and fulfill the requirements specified there.

### Measurement of Diameter

6. In each of the three procedures referred to in Section 5 the horizontal diameter shall be measured unless the particles are very uniform in shape, and in that event a diameter which most nearly represents the average shall be selected. For irregularly shaped particles, the horizontal diameter splitting the particle in half in one direction consistently maintained has been shown<sup>9</sup> to give fairly consistent and accurate results. For flat plates, it is necessary to consider the third dimension for accurate results. In any case, in reporting results, the diameter measured shall be specified.

<sup>7</sup> Henry Green, *Journal*, Franklin Inst., Vol. 192, p. 637 (1921).

<sup>8</sup> Sheppard and Lambert—not yet published.

<sup>9</sup> Martin, *Transactions*, Am. Ceramic Soc., Vol. 23, p. 61 (1924).

### Scale Limits for Measurement

7. (a) If more than 10 per cent by weight is coarser than 6.00 microns, the sample shall be elutriated prior to microscopic measurement. A graded scale for microscopic sizing is as follows:

Diameter Range in Microns	
General Case (20 per cent or more under 6 microns)	Special Case (Less than 20 per cent under 6 microns)
72 to 60	
60 to 48	
48 to 36	
36 to 24	
24 to 18	
18 to 12	
12 to 9	
9 to 6	
6.00 to 5.00	Same Scale and 6 to 4.5
5.00 to 4.00	4.5 to 3
4.0 to 3.5	3 to 1.5
3.5 to 3.0	1.5 to 0
3.0 to 2.5	
2.5 to 2.0	
2.0 to 1.8	
1.8 to 1.6	
1.6 to 1.4	
1.4 to 1.2	
1.2 to 1.0	
1.0 to 0.9	
0.9 to 0.8	
0.8 to 0.7	
0.7 to 0.6	
0.6 to 0.5	
0.5 to 0.4	
0.4 to 0.3	
0.3 to 0.2	
0.2	

(b) Two hundred and fifty particles shall be measured in each of three fields taken at random.

(c) The magnification shall be dependent on the class size being measured, and shall be sufficient to permit the differentiation specified above.

### Expression of Results

8. Results shall be expressed using one or more of the following average diameters:

Average Diameter	To Be Used:	Formula
$d_1$	As arithmetical mean.....	$d_1 = \frac{\sum nd}{\sum n}$
$\bar{d}_2$	To determine $S$ when $\rho$ is known	$\bar{d}_2 = \frac{\sum nd^3}{\sum nd^2}$

Average Diameter	To Be Used:	Formula
$\bar{d}_4$	To determine volume or weight distribution....	$\bar{d}_4 = \frac{\sum n d^4}{\sum n d^3}$
$\bar{D}$	To determine $N$ when $\rho$ is known	$\bar{D} = \sqrt[3]{\frac{\sum n d^3}{\sum n}}$
$\Delta$	To determine $S$ when $N$ is known	$\Delta = \sqrt{\frac{\sum n d^2}{\sum n}}$
$M_g$	As geometric mean or median.....	$\log M_g = \frac{\sum n \log d}{\sum n}$

## SYMBOLS:

$S_m$  = specific surface in square meters per gram,

$S_v$  = specific surface in square meters per cubic centimeter,

$N_m$  = number of particles per gram,

$N_v$  = number of particles per cubic centimeter,

$\sum n$  = number of particles in any given mass,

$\rho$  = density of the material,

$\theta$  = arithmetic standard deviation,

$\theta_g$  = geometric standard deviation, and

P.E. = probable error.

## Report

9. (a) If 90 per cent by weight fall in

any one class size, a frequency curve shall be given.

(b) In the 0 to 6-micron class, either a frequency curve or the distribution by weight shall be reported.

(c) In elutriated products, weight distribution shall be expressed and reported as follows: Weight distribution shall be the basis of comparison for all elutriated samples. The weight distribution for each elutriated portion shall be calculated on the basis of spherical particles, using the arithmetic mean of the microscopic class interval as the average diameter for all particles in that range. Each of the elutriated fractions shall total in weight distribution to the percentage retained in the elutriator and the composite curve shall be a summation of the distribution values in each range. The degree of overlapping is a measure of the efficiency of the elutriation, and it shall not be abnormal in amount.

# *Tentative Method for*

## DETERMINATION OF THE pH OF AQUEOUS SOLUTIONS WITH THE GLASS ELECTRODE<sup>1</sup>



A.S.T.M. Designation: E 70 - 46 T

ISSUED, 1946.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This method covers the apparatus and procedure for the electrometric measurement of pH values of aqueous or partly nonaqueous solutions or extracts with the glass electrode. It does not deal with the manner in which the solutions or extracts are prepared.

### Description of Terms

2. For the purpose of this method the term "meter" shall apply to the instrument used for the measurement of voltage (either in millivolts or in terms of pH units); the term "electrodes," to the glass and the reference electrode; and the term "assembly," to the combination of the meter and the electrodes. The performance of the meter shall be differentiated from that of the electrodes.

### Standard Buffer Solutions

3. (a) The buffer solutions for checking pH assemblies and glass electrodes shall be stored in bottles of chemically

resistant glass<sup>3</sup> and shall be prepared from salts (Note 1) sold specifically for use, either singly or in combination, as pH standards. The salts shall be dried for 1 hr. at 110 C. before use, except in the case of borax which shall be sold and used as the decahydrate. The buffer solutions shall be prepared in accordance with Paragraphs (b) to (f). Fresh buffer solutions shall be prepared at intervals not exceeding 3 months. The pH values of these buffer solutions at various temperatures are given in Table I.

NOTE 1.—Buffer salts of requisite purity can be obtained in the form of National Bureau of Standards standard samples, as follows:

Buffer Salt	Standard Sample Number
Acid potassium phthalate.....	185
Potassium dihydrogen phosphate.....	186-I
Disodium hydrogen phosphate.....	186-II
Borax .....	187

(b) *Distilled Water*.—Distilled water shall be boiled thoroughly, or purged with carbon dioxide-free air, to remove carbon dioxide and shall be protected

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Accepted by the Administrative Committee on Standards, December 5, 1946.

<sup>3</sup> Pyrex glass bottles have been found satisfactory for storage of the solutions.

with a soda-lime tube (Note 2) or the equivalent while cooling and in storage. The pH of this water shall be between 6.2 and 7.2 at 25 C. The specific conductance at 25 C. of the distilled water shall be not over  $2 \times 10^{-6}$  per ohm cm., and the residue on evaporation, when heated for 1 hr., shall be not more than 0.5 mg. per liter.

NOTE 2.—Precautions shall be taken to prevent mechanical contamination of the distilled water with traces of the material used for protection against carbon dioxide.

TABLE I.—pH VALUES FOR STANDARD BUFFER SOLUTIONS FROM 0 TO 60 C.<sup>a</sup>

Temperature, deg. Cent.	0.1 N Hydro- chloric Acid	0.05 M Phthalate	0.025 M Phosphate	0.01 M Borax
0.....	1.10	4.01	6.98	9.46
5.....	1.10	4.01	6.95	9.39
10.....	1.10	4.00	6.92	9.33
15.....	1.10	4.00	6.90	9.27
20.....	1.10	4.00	6.88	9.22
25.....	1.10	4.01	6.86	9.18
30.....	1.10	4.01	6.85	9.14
35.....	1.10	4.02	6.84	9.10
40.....	1.10	4.03	6.84	9.07
45.....	1.10	4.04	6.83	9.04
50.....	1.11	4.06	6.83	9.01
55.....	1.11	4.08	6.84	8.99
60.....	1.11	4.10	6.84	8.96

<sup>a</sup> For a discussion of the manner in which these pH values were assigned, see the paper by G. G. Manov, "Standard Buffer Solutions," Symposium on pH Measurement, *Proceedings, Am. Soc. Testing Mats.*, Vol. 46 (1946).

(c) *Hydrochloric Acid Buffer Solution* (0.1 N, pH 1.10 at 25 C.)—Dilute hydrochloric acid, conforming to the specifications of the American Chemical Society, with the distilled water specified in Paragraph (b) to form a  $0.1000 \pm 0.0005$  N solution. Standardize this solution against a carbonate-free solution of sodium hydroxide of known strength that has been standardized against National Bureau of Standards standard sample No. 84c of acid potassium phthalate.

(d) *Acid Potassium Phthalate Buffer Solution* (0.05 M, pH 4.01 at 25 C.)—Dissolve  $10.21 \pm 0.05$  g. of acid potas-

sium phthalate in distilled water and dilute to 1 liter with distilled water in a volumetric flask.

(e) *Phosphate Buffer Solution* (0.025 M, pH 6.86 at 25 C.)—Dissolve  $3.402 \pm 0.001$  g. of potassium dihydrogen phosphate and  $3.549 \pm 0.001$  g. of disodium hydrogen phosphate in distilled water and dilute to 1 liter with distilled water in a volumetric flask.

(f) *Borax Buffer Solution* (0.01 M, pH 9.18 at 25 C.)—Dissolve  $3.81 \pm 0.01$  g. of decahydrated disodium tetraborate in distilled water and dilute to 1 liter with distilled water in a volumetric flask. Keep the bottle containing this solution protected from atmospheric carbon dioxide or stopper the bottle at all times except when actually in use.

### Apparatus

4. The apparatus shall consist of the following:

(a) *Meter*.—There shall be three permissible types of meters (types I, II, and III), the particular choice to be made depending upon the desired or specified accuracy in the determination. For routine determinations of pH, meters of types I, II, or III may be used. For referee work or in case of dispute, type I meters shall be used. These designations of types of meters are made on the basis of the electrical characteristics of the meter and of the operating performance of the assembly. The requirements for the electrical characteristics of meters of types I, II, and III and the operating performance of the assemblies are given in Table II. The electrical shielding shall be sufficient to avoid the effects of body capacitance.

(b) *Glass Electrode*.—The composition of the glass electrode (Note) shall be such that the stability and pH response shall conform to the requirements pre-

scribed in Section 5 (b). Glass electrodes for use with meters of types I and II shall have a resistance of approximately 500 megohms at 25 C., and electrodes for use with type III meters shall have a resistance of not over 10 megohms. The leads of glass electrodes intended for use outside the electrode compartment of the meter (external

all times there shall be an excess of solid potassium chloride surrounding the calomel. For routine test work, the solution surrounding the calomel may be saturated at 0 C. with potassium chloride. The junction between the saturated potassium chloride solution of the reference electrode and the buffer or test solution shall permit of easy washing

TABLE II.—ELECTRICAL CHARACTERISTICS OF METERS AND OPERATING PERFORMANCE OF ASSEMBLIES.

	Type I Meter	Type II Meter	Type III Meter
ELECTRICAL CHARACTERISTICS OF METERS			
Vacuum-tube operation.....	yes	yes	no
Type of measuring circuit.....	potentiometric	direct reading	potentiometric
Method for detection of balance point.....	null indicator (galvanometer or electron-ray tube)	direct reading	lamp and scale galvanometer <sup>a</sup>
Maximum grid current in amperes drawn from glass electrode during measurement of pH or electromotive force.....	$2 \times 10^{-12}$	$5 \times 10^{-11}$	
Standard cell for calibration of working battery.....	yes	optional	yes
Scale:			
Units shown.....	millivolts or pH or both	pH	millivolts or pH or both
Minimum range in pH units.....	0 to 13	0 to 13 <sup>b</sup>	0 to 13
Minimum range in millivolts.....	0 to $\pm 1100$		0 to $\pm 1100$
Maximum value of smallest ruled interval.....	0.1 pH 10 mv.	0.1 pH	0.1 pH 10 mv.
Asymmetry potential compensator.....	yes	yes	optional
Automatic or manual temperature compensator.....	yes	optional <sup>c</sup>	optional <sup>c</sup>
Minimum range, deg. Cent.....	10 to 40	10 to 40	10 to 40
Maximum value of smallest graduation, deg. Cent.....	1	1	1
Power supply, batteries or 110-volt a.c.....	either	either	batteries
OPERATING PERFORMANCE OF ASSEMBLIES			
Maximum error at upper end of scale between applied and observed voltage (see Section 5).....	$\pm 2$ mv.	$\pm 6$ mv.	$\pm 6$ mv.
Reproducibility on buffered solutions.....	$\pm 0.02$ pH	$\pm 0.05$ pH	$\pm 0.05$ pH
Overall accuracy between pH 1 and 12, exclusive of sodium-ion error of electrode.....	$\pm 0.05$ pH	$\pm 0.1$ pH	$\pm 0.1$ pH

<sup>a</sup> Minimum sensitivity equivalent to 0.0005 microamperes per mm. at one meter.

<sup>b</sup> A double scale may be provided.

<sup>c</sup> If a temperature compensator is not provided, the instrument shall be furnished with suitable charts or graphs giving corrections for every degree from 10 to 40 C. for various pH readings.

electrodes) shall be sufficiently well shielded to avoid the effects of body capacitance.

NOTE.—Requirements for the physical dimensions and shape of the electrodes and the composition of the internal reference solution are not considered part of this method.

(c) *Reference Electrode*.—For referee purposes, the saturated calomel electrode shall be the reference electrode, and at

to remove traces of solution from the previous determination and shall be of such construction that a fresh junction can be formed for each test.

(d) *Storage of Electrodes*.—The lower halves of the glass and calomel electrodes shall be stored in distilled water when not in use. The "low sodium-error" electrodes shall be stored in a buffer solution of approximately pH 10.



### Performance Tests of Meter and Electrodes

NOTE.—Except for referee or research purposes it will usually be unnecessary to perform the tests indicated in Section 5. For routine work the standardization of the assembly described in Section 6 will suffice.

5. (a) *Meter*.—The meter shall be brought to electrical balance in accordance with the manufacturer's instructions. The performance shall then be tested applying a known, variable potential through a resistance of approximately 500 megohms (10 megohms in the case of type III meters) to the terminals of the meter, the high-resistance lead being connected to the terminal corresponding to the glass electrode. (The source of known potential may be a precision-type potentiometer with a range of 1100 mv. or more and a limit of error not greater than 0.1 mv. The 500 (or the 10) megohm resistor shall be properly shielded to avoid capacity pickup. The general precautions to be observed are those customary in measurements of high impedances.) Commencing with a value of zero, the applied potential shall be increased in increments of 100 mv., and the readings of the dial of the meter at balance shall be noted. The process shall be completed to cover the entire range of the meter. If the meter is calibrated in terms of pH only, the observed differences in pH shall be multiplied by 59.1 to convert to millivolts. In no case shall the difference between the applied voltage and that indicated on the meter differ by more than 1 mv. per increment of applied voltage, and the cumulative error at the end of the scale shall not be more than plus or minus 2 mv. for type I meters and plus or minus 6 mv. for meters of types II and III.

(b) *Glass Electrodes*.—The difference in the potentials between the glass and the hydrogen electrodes shall be meas-

ured when both electrodes are immersed in the identical portion of various buffer solutions over the range of pH of approximately 1 to 13. The cell used for these comparisons shall be placed in a water bath thermostatically controlled to within plus or minus 0.1 C. at any convenient temperature between 20 and 30 C. The buffers used for this test shall be those listed in Section 3. In addition, the following buffer solutions shall also be used: (1) a phosphate buffer solution, approximate pH 11.7 at 25 C., prepared by dissolving  $2.556 \pm 0.001$  g. of dried disodium hydrogen phosphate in  $138.0 \pm 0.5$  ml. of carbonate-free 0.1 *M* sodium hydroxide and diluting to a total volume of 1 liter; and (2) a carbonate-free solution of sodium hydroxide, which has concentration of  $0.1000 \pm 0.0005$  *M* and an approximate pH value of 12.9. The difference in the potentials between the glass electrode and hydrogen electrode shall be independent to within 0.02 unit of the pH of the solution up to pH 9.18 for electrodes of all types and not more than 0.1 unit at pH 12.9 for electrodes of the "low sodium-error" type.

### Standardization of the Assembly

6. (a) The temperature control knob, if one is provided, shall be set to correspond to the proper temperature. The instrument shall be turned on, allowed to warm up thoroughly, and brought to electrical balance by carefully following the manufacturer's instructions. The glass electrode shall be thoroughly washed with distilled water (Note 1). If the calomel electrode is of the enclosed or pencil type in which contact of the saturated potassium chloride solution is made through a capillary, it shall be washed in the same manner and at the same time as the glass electrode. If the calomel electrode is of the type in which the saturated potassium chloride solution has free access to the buffer

solution, the old junction shall be flushed and a fresh one formed.

NOTE 1.—It is suggested that the electrodes be given at least five washings of approximately 15 ml. each with a stream of water from a wash bottle, followed by two additional washings of approximately 50 ml. each from the mouthpiece of the wash bottle. The sample cup should be similarly washed and shaken to remove the excess drops of water.

(b) The sample cup shall then be filled with phthalate buffer solution and the electrodes immersed in the cup. In type I meters the dial of the instrument shall be set to read the pH value appropriate to the standardizing buffer solution used. The operating button shall be engaged and the zero control (sometimes called the asymmetry potential) knob rotated until the meter is brought to balance. In type II meters the operating button shall be engaged and the zero control knob rotated until the reading of the dial corresponds to the known pH of the standardizing buffer solution. Type III meters usually have no zero adjustment, but the value for the potential of the cell combination should be noted. The process shall then be repeated, using additional portions of the standardizing buffer solution, until a reproducibility is reached of plus or minus 0.02 unit for type I meters, 0.05 unit for type II meters, and 0.1 unit for type III meters. The electrodes shall then be washed off as described in Paragraph (a).

(c) The second buffer solution (Note 2) selected from Table I for the standardization of the assembly shall correspond as closely as possible to the anticipated value for the solution the pH of which is to be determined. The above process shall be repeated except that the zero control knob shall not be adjusted for the standardization against the second buffer solution. The assembly shall be judged to be operating

satisfactorily if the reading obtained for the second buffer solution agrees with the known value of that buffer solution to within 0.02 unit for type I meters, 0.05 unit for type II meters, and 0.1 unit for type III meters. The electrodes shall then be washed as described in Paragraph (a). The meter now shall be considered ready for the determination of the pH of a test solution.

NOTE 2.—The assembly shall always be calibrated with two buffer solutions to check the linearity of the response of the electrode at different pH values and to detect a faulty glass electrode. The standardization of the assembly with only a single buffer solution may be completely erroneous because of the wide range of the zero control knob. The presence of a faulty electrode will be detected by failure to obtain a reasonably correct value (plus or minus 0.02 unit) for the pH of the second buffer solution when the meter has been standardized in terms of the first. A cracked electrode will yield pH values that are essentially the same for both buffer solutions.

(d) The assembly should be calibrated every time it is used, if only an occasional pH determination is made, and at short convenient intervals, for example, every fifth sample, in a long series of measurements. The calibration with standard buffer solutions shall be performed at both the beginning and the end of the measurements.

#### Determination of pH of Test Solution

7. (a) If a precision of greater than 0.1 pH unit is desired, the temperature of the standardizing buffer solutions, the glass and calomel electrodes, and the test solution shall be within 2 C. of one another. Electrodes, buffer solutions, test solutions, and wash water shall be kept at the temperature of measurement for at least 2 hr. prior to making the measurement in order to reduce to a negligible value the effects of thermal or electrical hysteresis of the electrodes.

(b) After the meter has been calibrated with two standard buffer solu-

tions (Section 6), the electrodes and the sample cup shall be thoroughly washed as described in Section 6 (a). The cup shall then be filled with a portion of the test solution and a preliminary value obtained for its pH. In general, this value will drift and should be regarded as an estimate. Subsequent readings taken on additional portions of the same test solution will yield successively more constant pH values. In the case of test solutions that are well buffered, three portions may be sufficient to yield pH values that are reproducible to within plus or minus 0.02 unit and that show drifts of less than plus or minus 0.02 unit in 1 or 2 min. The pH of very dilute or unbuffered solutions shall be measured in the manner directed above, except that the solution shall be placed in a suitable wide-mouth flask that previously has been flushed with carbon dioxide-free air. The contents of the flask shall be protected from exposure to atmospheric carbon dioxide. The pH values of such solutions may be reproducible to approximately 0.05 unit.

(c) The pH of solutions more alkaline than pH 9 may also be determined with the use of the "low sodium-error" glass electrodes. The manufacturer's instructions shall be followed carefully.

(d) The pH of distilled water shall be measured by first calibrating the assembly with phthalate buffer and making measurements of the pH of the distilled water in a carbon dioxide-free atmosphere as directed in Paragraph (b), and then recalibrating the assembly with borax buffer and making a second set of measurements of the pH of the distilled water. In this manner two values will result, the first of which will be slightly more acid and the second slightly more alkaline than the true pH of the water. The two values, however, should check to within 0.1 unit and the average shall be reported. The "flow-type" electrodes may also be used.

#### Report

8. (a) The pH values obtained for the solution shall be reported to the nearest 0.05 or 0.1 unit, depending on the type of the meter used and the precision set forth in the specifications for the material under test.

(b) The pH of solutions that are only partially aqueous in character shall be approximated in the manner described in Section 7. The values so obtained shall be reported as apparent pH values. Particular care should be taken to keep the electrodes free of deposits of foreign matter.

# *Tentative Method of Test for*

## SOFTENING POINT<sup>1</sup>

### BALL AND SHOULDERED RING APPARATUS



A.S.T.M. Designation: E 28 - 42 T

ISSUED, 1936; REVISED, 1939, 1942.<sup>2</sup>

This Tentative Method has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

#### Scope

1. (a) This method of test for softening point by ring-and-ball method is applicable to asphalts, tars, pitches, rosins, and most resins both natural and synthetic.

(b) *Softening Point*.—In general, with materials of this type, softening does not take place at a definite temperature. As the temperature rises, these materials gradually and imperceptibly change from brittle or exceedingly thick and slow flowing materials to softer and less viscous liquids. For this reason the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

#### Apparatus

2. The apparatus shall consist of the following:

(a) *Ring*.—A brass shouldered ring conforming to the dimensions (Note) shown in Fig. 1 (a). The ring may be attached, by brazing, to a No. 11 B. & S. gage brass wire (diameter 2.31 mm. = 0.091 in.), (see Fig. 2).

NOTE.—The tapered ring formerly specified in the Tentative Method of Test for Softening Point (Ball and Tapered Ring Apparatus) (A.S.T.M. Designation: E 28 - 39 T) of the American Society for Testing Materials,<sup>3</sup> may be used interchangeably with the shouldered ring.

(b) *Ball*.—A steel ball, 9.53 mm. ( $\frac{3}{8}$  in.) in diameter weighing between 3.45 and 3.55 g.

(c) *Ball Guide*.—A ball guide for centering the balls, constructed of brass, and having the shape and dimensions illustrated in Fig. 1 (c).

(d) *Container*.—A glass vessel, capable of being heated, not less than 8.5 cm. ( $3\frac{1}{2}$  in.) in diameter and not less than 10.5 cm. ( $4\frac{1}{8}$  in.) in depth from the bottom of the flare. (A 600-ml. low-form Griffin beaker meets this require-

<sup>1</sup> Under the standardization procedure of the Society, this method is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> The ring-and-ball method was first standardized by the Society's Committee D-4 on Road and Paving Materials as applying to bituminous materials and issued under the A.S.T.M. Designation D 36. The present method is an extension and modification of the ring-and-ball method to make it applicable to materials other than bituminous materials as well.

<sup>3</sup> 1939 Book of A.S.T.M. Standards, Part II, p. 1169; Part III, p. 1127.

ment. For use with the ring holder an 800-ml. tall-form beaker is convenient.)

(e) *Support for Ring and Thermometer.*—Any convenient method for supporting the ring and thermometer may be used (see Figs. 2, 3, and 4) provided it meets the following requirements: The ring shall be supported in a substantially

the upper surface of the lower horizontal plate; the lower horizontal plate shall be at least 1.3 cm. ( $\frac{1}{2}$  in.) above the bottom of the glass vessel. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.0 cm. ( $\frac{3}{8}$  in.) but not touching the ring. For

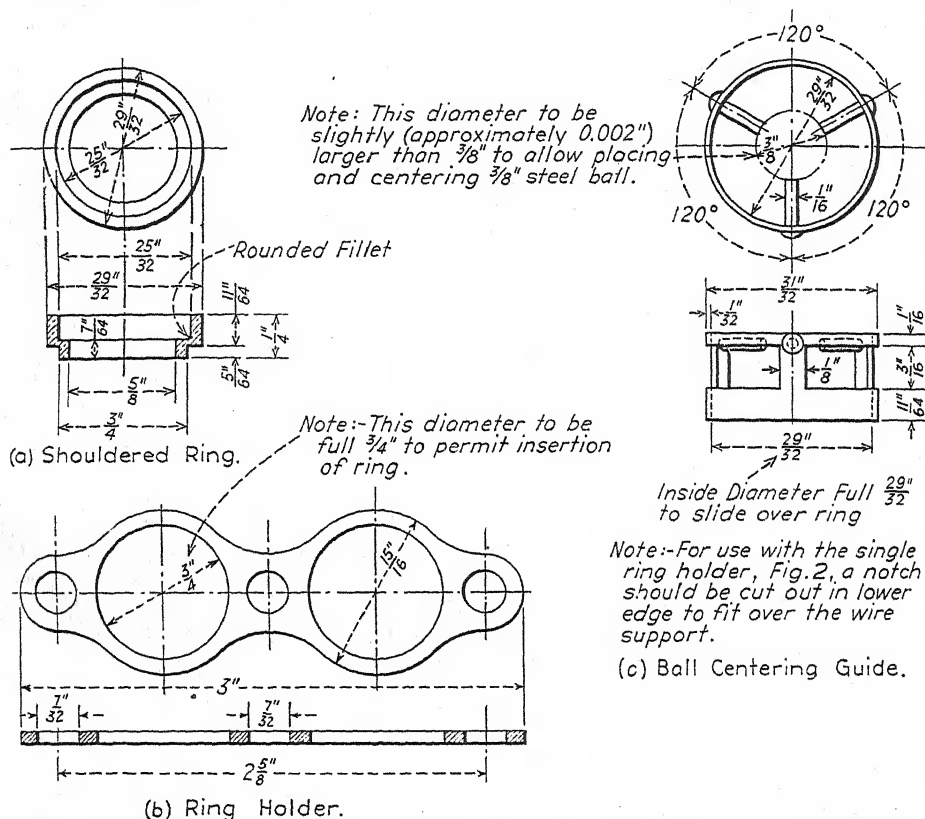


FIG. 1.—Shouldered Ring, Ring Holder, and Ball-Centering Guide.

horizontal position. The top of the ring shall be at least 7.6 cm. (3.0 in.) below the top of the container and at least 5.1 cm. (2.0 in.) below the surface of the heating liquid. Using the apparatus in Fig. 1, the bottom of the ring shall be 2.5 cm. (1.0 in.) above the bottom of the glass vessel. If the ring holder is used (see Fig. 3), the bottom of the ring shall be 2.5 cm. (1.0 in.) above

referee work the ring holder shall not carry more than two rings.

(f) *Thermometers.*—(1) An A.S.T.M. Low Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of  $-2$  to  $+80$  C. or  $+30$  to  $+180$  F. and conforming to the requirements for thermometer 15C-39 or 15F-39, respectively, as prescribed in the Standard

Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.<sup>4</sup>

(2) An A.S.T.M. High Softening Point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 30 to 200 C.

a manner as will avoid the inclusion of the surface layer. A quantity at least twice that necessary to fill the desired number of rings, and in no case less than 40 g., shall be melted immediately in a clean container, using an oven, hot plate, sand or oil bath to prevent local

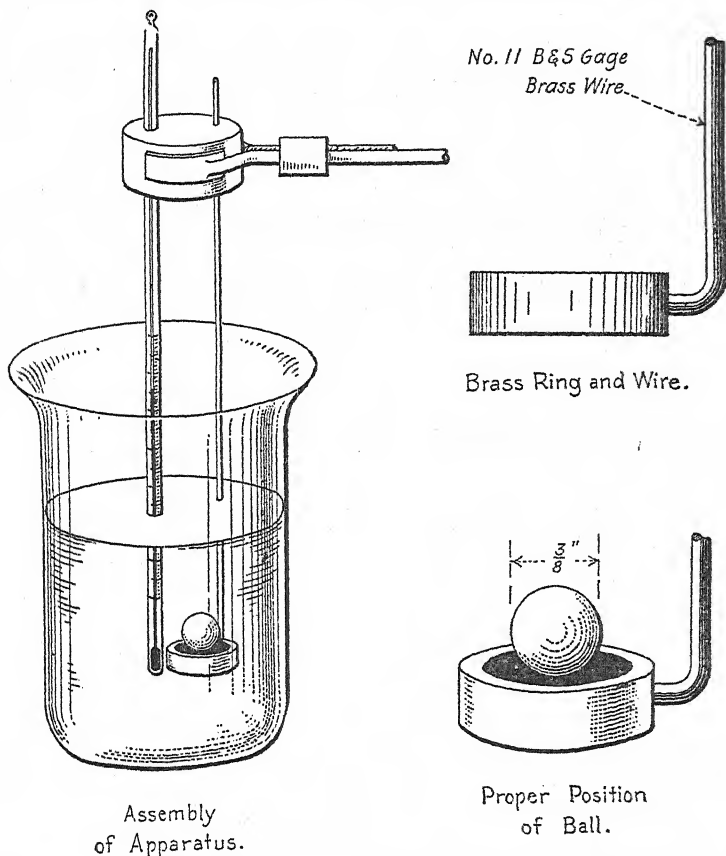


FIG. 2.—Assembly of Apparatus, Showing Single Shouldered Ring.

or 85 to 392 F. and conforming to the requirements for thermometer 16C - 39 or 16F - 39, respectively, as prescribed in Standard Specifications E 1.<sup>4</sup>

#### Preparation of Sample

3. (a) The sample shall be selected from the material to be tested in such

overheating. Care shall be taken to avoid incorporating air bubbles in the sample which shall not be heated above the temperature necessary to pour the material readily without inclusion of air bubbles. The time from the beginning of heating to the pouring of the sample shall not exceed 15 min.

(b) Immediately before filling the rings, they shall be preheated to ap-

<sup>4</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

proximately the temperature at which the material is to be poured. The rings, while being filled, should rest on an amalgamated brass plate. The quantity of material poured into the rings shall be such that after 20-min. cooling at room temperature of materials with softening points below 80 C. (176 F.) and 40-min. cooling for materials of higher softening point, an excess amount will remain which shall then be cut off cleanly with a slightly heated spatula. For routine testing this period of time may be curtailed according to the characteristics of the material

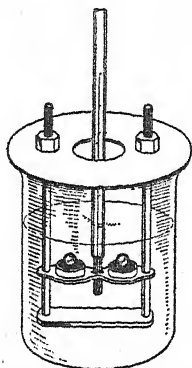


FIG. 3.—Assembly of Apparatus, Showing Two Rings.

under test. In case the test is to be repeated, a clean container and fresh sample shall be used.

#### Procedure

4. (a) *Assembling Apparatus.*—The glass container shall be filled to a depth not less than 9 cm. (3.5 in.) with freshly boiled distilled water (or with glycerol for materials melting above 80 C. (176 F.)) which has been cooled to not less than 45 C. (113 F.) below the anticipated softening point but in no case lower than 5 C. (41 F.) for materials melting below 80 C. (176 F.) and in no case lower than 35 C. (95 F.) for materials melting above 80 C. (176 F.). Provision shall

be made for centering the ball on the upper surface of the sample, either by use of the ball guide or by making a slight indentation in the center of the sample. The latter may be done by pressing the ball or a rounded rod into the material at this point. In the case of hard materials the rod may be heated. The ring containing the sample and ball guide, if used, shall be suspended in the water so that the lower surface of the

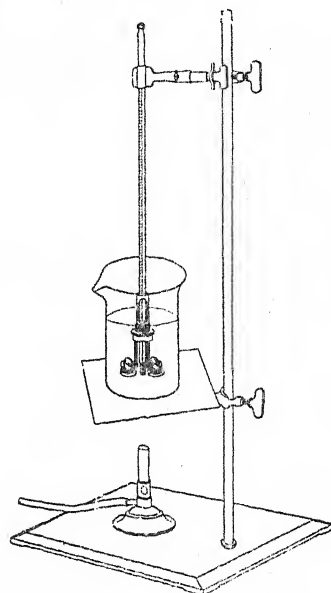


FIG. 4.—Assembly of Apparatus, Showing Four Rings.

filled ring is 2.5 cm. (1 in.) above the bottom of the glass container (Fig. 2) or 2.5 cm. above the upper surface of the lower horizontal plate which is at least 1.3 cm. ( $\frac{1}{2}$  in.) above the bottom of the glass vessel (Fig. 3) and so that its upper surface is at least 5.1 cm. (2 in.) below the surface of the water. The ball shall be placed in the water but not on the specimen. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.0 cm. ( $\frac{3}{8}$  in.) but not touch-



ing the ring. The initial temperature shall be maintained for 15 min. With suitable forceps the ball shall be placed in the center of the upper surface of the material in the ring.

(b) *Heating*.—To facilitate uniform heating, the burner shall be placed midway between the center and the edge of the beaker on a diameter at right angles to the diameter which includes the ring or rings and the thermometer bulb. The effect of drafts on uniformity of heating must be eliminated, using shields if necessary. The heat shall be applied in such manner that the temperature of the bath is raised 5 C. (9 F.) each minute.

(c) *Permissible Variation in Rise of Temperature*.—The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be plus or minus 0.5 C. (0.9 F.). All tests in which the rate of rise exceeds these limits shall be rejected.

#### Softening Point

5. (a) The temperature reading to one-half of the smallest scale division indicated by the thermometer at the instant the sample touches the bottom of the container (or the lower horizontal plate) shall be taken as the softening

point. No corrections shall be made for emergent stem of the thermometer.

(b) For materials softening around 80 C. (176 F.) the nature of the bath fluid (water or glycerol) shall be reported, since a glycerol bath yields slightly higher results than a water bath.

#### Precautions

6. (a) The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and affect the results. Rigid adherence to the prescribed rate of heating is absolutely essential for reproducibility of results.

(b) A thin, amalgamated copper plate or a sheet of filter paper placed on the bottom of the glass vessel will prevent the material from sticking to the bottom of the glass vessel, thereby saving considerable time and trouble in cleaning.

(c) In order to insure uniform heat distribution at all times throughout the bath, a mechanical stirrer should be used.

#### Reproducibility of Results

7. With care and proper attention to details, duplicate determinations of softening point by this method should not differ by more than 1.0 C. (1.8 F.).

# *Tentative Recommended Practice for*

## CHARACTERISTICS OF STANDARD CARBON ARC ACCELERATED WEATHERING UNIT<sup>1</sup>



A.S.T.M. Designation: E 42 - 42 T

ISSUED, 1942.<sup>2</sup>

This Tentative Recommended Practice has been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

### Scope

1. This recommended practice covers the basic principles of an accelerated weathering unit of the carbon arc type.

NOTE.—Inasmuch as weather varies from day to day and place to place, thus making it impossible to correlate with the weather at all times and for all locations, it is desirable to promote the adoption of such cycles as will reproduce the particular weathering influences that are of interest. It is believed that the characteristics recommended herein cover those points of fundamental importance that will provide an acceptable accelerated weathering unit for many purposes, and at the same time permit the development of detailed and specific cycles needed for the obtaining of particular data and falling within the general structure of this recommended practice. As an example, if the emphasis is on rust-inhibiting paint, in addition to any cycle adopted, it may be desirable to have an accessory high humidity cabinet made available.

### Apparatus

2. (a) The light source shall be a carbon arc.

(b) The carbons shall be of such com-

position and operating under such conditions that the quality of the spectrum as it strikes the test specimens shall approach so far as possible the spectrum of the sun.

(c) The weathering unit shall include means for measuring and controlling the following:

- (1) Current,
- (2) Voltage,
- (3) Temperature of air and water, and
- (4) Running time.

(d) The materials of construction shall be of such character as not to react with the test specimens during the test.

### Procedure

3. (a) Light values shall be measured by the oxalic acid - uranyl sulfate method described in the Tentative Method of Calibrating a Light Source Used for Accelerating the Deterioration of Rubber (A.S.T.M. Designation: D 749).<sup>3</sup>

(b) Specimens shall be mounted vertically and shall rotate about the arc in order to provide uniform distribution of the light. If the specimens are mounted

<sup>1</sup> Under the standardization procedure of the Society, this recommended practice is under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Accepted by Committee E-10 on Standards, August 24, 1942.

<sup>3</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.

vertically both above and below the horizontal center line of the light source, their position should be transposed periodically to provide uniform distribution of the light in a vertical plane over the entire face of the specimen. Products of combustion shall not be permitted to touch the specimens.

(c) The air temperature at the distance of the surface of the specimens under test shall be constant. It shall be determined by a shielded thermometer at a point most remote from the water spray.

(d) Water sprays shall be provided whereby clean water such as drinking water may be forced on the specimens to simulate the washing action of rain,

to provide moisture for causing alternate expansion and contraction due to swelling and drying out, and to introduce thermal shock and sweating. No recirculation or immersion in the spray water shall be permitted.

NOTE.—Certain types of water may contain constituents deleterious to the materials under test.

(e) The accelerated weathering unit at all times shall be operated under constant voltage at the recommended arc amperage. The proper carbons shall be used, the globes and filters cleaned at frequent intervals, and the temperatures of the air and water shall be regulated within the tolerances established.

*Tentative Recommended Practices for*  
DESIGNATION OF NUMERICAL REQUIREMENTS IN  
STANDARDS<sup>1</sup>



A.S.T.M. Designation: E 29 - 40 T

ISSUED, 1940.<sup>2</sup>

These Tentative Recommended Practices have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

**Scope**

1. (a) These recommended practices are intended to assist the various standing committees in the use of uniform terms and conventions in expressing numerical requirements in specifications and methods of testing. Their aim is to outline practices which should aid in clarifying the intended meaning of limiting values, with which test values are compared in acceptance and rejection of material. Certain nonnumerical terms closely related to numerical expressions are also included.

(b) These practices are not intended to be used in the interpretation of specifications which do not definitely call for their use. The provisions of these practices apply to the Society's standards as they bear on the acceptance and rejection of material, however, when such standards make definite reference to the practices as covered in Section 2.

(c) Definite interpretations are assigned to the following:

(1) The phrase "to the nearest ....." when applied to a specified limit (see Section 4 (a)).

(2) The underlining of figures in a specified limit, for example, "1.500" (see Section 5 (a)).

(3) The symbol " $\pm$ " when applied to a specified value of maximum permissible deviation (see Section 8 (b)).

(4) The term "average" (see Section 9 (a)).

**Reference to These Practices in Standards**

2. The inclusion of the following paragraph, or a suitable equivalent, in any standard (preferably immediately after the section on "Scope") shall constitute due notification that numerical requirements in that standard are subject to the interpretations set forth in these recommended practices:

*Interpretation of Numerical Requirements.*—The numerical requirements of this standard are subject to the interpretations set forth in the

<sup>1</sup> Under the standardization procedure of the Society, these recommended practices are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Accepted by the Society at annual meeting, June, 1940.

Tentative Recommended Practices for Designation of Numerical Requirements in Standards (A.S.T.M. Designation: E 29) of the American Society for Testing Materials. Particular attention is called to the use of—(insert one of the following: (1) the phrase “to the nearest .....”; (2) underlining; (3) underlining and use of the phrase “to the nearest .....”)—in expressing specified limits.

### Expression of Numerical Requirements

3. (a) The unqualified statement of a numerical limit, such as “2.50 in. max.”, cannot, in view of different established practices and customs, be regarded as carrying a definite operational meaning (1) as to the number of places of figures to be read on the scale of measurement, or (2) as to the number of places of figures to be retained in an observed or calculated value for purposes of acceptance and rejection.

**Measurement.**—In some fields, specified values of 2.5 in. max., 2.50 in. max., 2.500 in. max., are taken to imply that a length determination should be made to the nearest 0.1 in., 0.01 in., 0.001 in., respectively. In other fields, no such significance is attached to this difference of expression.

**Acceptance and Rejection.**—In some fields, specified values of 2.5 in. max., 2.50 in. max., 2.500 in. max., are taken to imply that for purposes of acceptance and rejection, observed values or calculated values should be rounded off to the nearest 0.1 in., 0.01 in., 0.001 in., respectively. In other fields, these three specified values are taken to imply the same absolute limit of two and one-half inches and that no deviation, however small, in excess of this value is allowed.

(b) Clear meaning on these points can be assured only by specifying the following operations:

(A) *How measurement values are to be obtained, and*

(B) *How the observed values or calculated values are to be handled when they are compared to specified values in determining acceptability of material.*

(c) In specifying operation (A) the following items should be included:

(1) The test or analytical method to be followed,

(2) The type of measuring device to be used,

(3) The units of measurement to be used, and

(4) The number of places of figures to be read on the scale of measurement.

(d) Methods of specifying operation (B) are given in the following Paragraphs (e) to (g).

(e) The numerical requirements should be so expressed that the number of places of figures to be considered significant for purposes of acceptance and rejection are clearly indicated.

(f) If *all* places of figures in an observed or calculated value are to be considered significant for purposes of acceptance and rejection, this intent may be made clear by a direct statement in the standard, such as: “Observed or calculated values should not be rounded off but should be compared directly with specified values, for purposes of acceptance and rejection.”

(g) If a *limited* number of places of figures in an observed or calculated value are to be considered significant for purposes of acceptance and rejection, this intent may be expressed by the use of the phrase “to the nearest .....,” as follows:

Carbon (to the nearest 0.01 per cent).....	0.20 per cent, min.
Tensile strength (to the nearest 100 psi.).....	62 000 psi., min.

When the use of the phrase “to the nearest.....” is not practicable, the number of figures to be considered significant may be indicated by a direct statement or by underlining the significant places, as follows:

Carbon.....	0.20 per cent, min.
Tensile strength.....	<u>62 000</u> psi., min.

**Meaning of Phrase "to the nearest....."**

4. (a) The phrase, "to the nearest 10, 1, 0.1, 0.01, etc.," when applied to specified limits, shall be interpreted to have the meaning that an observed value or a calculated value is, for purposes of acceptance and rejection, to be rounded off to the nearest unit in the place occupied by the digit 1 in the phrase "to the nearest....."; that is, shall be rounded off to the nearest digit in the last right-hand place of figures considered significant.

Specified Limit		Observed Value or Calculated Value	Rounded-off Value to be Used for Acceptance Purposes	Conforming to Specified Limit
Tensile Strength (to the nearest 100 psi.)	60 000 psi., min.	59 940	59 900	No
		59 950	60 000	Yes
		59 960	60 000	Yes
Tensile Strength (to the nearest 100 psi.)	60 500 psi., min.	60 440	60 400	No
		60 450	60 400	No
		60 460	60 500	Yes
Antimony (to the nearest 0.01 per cent)	0.90 per cent, min.	0.894	0.89	No
		0.895	0.90	Yes
		0.896	0.90	Yes
Antimony (to the nearest 0.01 per cent)	0.91 per cent, min.	0.904	0.90	No
		0.905	0.90	No
		0.906	0.91	Yes

NOTE.—The final rounded-off value should be obtained from the most precise value available and not from a series of successive rounded-off values; for example, 59,490 psi. to the nearest 100 and then to the nearest 1000 psi. becomes 59,500 and 59,000 (not 60,000) psi., respectively, since the most precise value available is less than 59,500.

(b) The actual rounding-off procedure shall be carried out as follows:<sup>2</sup>

(1) When the figure next beyond the last figure or place to be retained is less than 5, the figure in the last place retained should be kept unchanged.

(2) When the figure next beyond the last figure or place to be retained is more than 5, the figure in the last place retained should be increased by 1.

(3) When the figure next beyond the last figure or place to be retained is 5, and

(a) there are no figures, or only zeros, beyond this 5, if the figure in the last place to be retained is odd, it should be increased by 1; if even, it should be kept unchanged;

(b) if the 5 next beyond the figure in the last place to be retained is followed by any figures other than zero, the figure in the last place retained should be increased by 1, whether odd or even.

The above rules for rounding off may be restated as follows:

(A) The figure in the last place to be retained should be kept unchanged (a) when the figure in the next place is less than 5; (b) when the figure in the next place is 5 followed by no other figures or by only zeros, and the figure in the last place retained is even.

(B) The figure in the last place to be retained should be increased by 1, (a) when the figure in the next place is more than 5; (b) when the figure in the next place is 5 followed by no other figures or by only zeros, and the figure in the last place retained is odd; (c) when the figure in the next place is 5, followed by any figure or figures other than zero.

(c) If it should be necessary to round off to the nearest 50, 5, 0.5, 0.05, etc., this can be done by the application of the same general principles used above except that in case of observed or calculated values that are exactly half-way between two rounded-off values, the rounding off is done step-wise, that is, in two successive steps; for example, 6025 to the nearest 50 first becomes 6020 (Section 4 (b), item (3) (a)), which in turn becomes 6000; while 6075 to the nearest 50 first becomes 6080 (Section 4 (b), item (3) (a)), which in turn becomes 6100.

**Meaning of the "Underlining" Symbol**

5. (a) Underlining, when applied to specified limits shall be interpreted to

<sup>2</sup> This rounding-off procedure agrees with that adopted in the American Standard Rules for Rounding Off Numerical Values (ASA No: Z25.1—1940).

have the meaning that an observed value or a calculated value is, for purposes of acceptance and rejection, to be rounded off to the nearest unit in the last right-hand place of figures underlined.

(b) Thus underlining is the simple equivalent of the phrase "to the nearest 10, 1, 0.1, 0.01, etc."

#### Use of the Phrase "to the nearest...." and of the Underlining Symbol

6. (a) The use of the phrase "to the nearest....." (or of underlining) in designating specified limits, in effect specifies that the operation of "rounding off" observed or calculated values shall be considered applicable in determining whether those values are conforming or nonconforming with the specified limit.

(b) It is recommended that the rounding-off method as indicated by the use of the phrase "to the nearest....." (or by underlining) be employed as widely as possible in designating limits for dimensions. In view of the fact that well established gage tolerances and other practices in some fields do not employ the principle of "rounding off" here discussed, the use of the phrase "to the nearest....." (or of underlining) may not be considered universally applicable to all dimensional requirements. Where other procedures are to be used, it is recommended that the standard contain a direct statement as to the practice intended by the committee originating the specification. Clear expression on this point should aid in preventing misunderstandings and disagreements in instances where observed values are marginally close to specified values.

(c) It is recommended that the phrase "to the nearest....." (or underlining) be used generally in designating percentage chemical composition requirements, and that the number of decimal places used in expressing limiting values be the same as the number of decimal places to be

considered significant for purposes of acceptance and rejection.

(d) In the interests of simplicity of expression, a general statement, used as a caption or a footnote, should be used in preference to repetitions of the phrase "to the nearest....." (or to repeated underlining) in instances where the "rounding-off" method is to be specified for a large number of similar requirements. Suggestions as to how this may be accomplished are given in the following examples:

(1) If the rounding-off method is to apply to all specified limits in the standard and if all expressed figures are to be considered significant, this may be indicated by adding the following sentence to the notification paragraph given in Section 2, as follows:

The following applies to all specified limits in this standard: For purposes of acceptance and rejection, an observed value or a calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value.

(2) If the rounding-off method is to apply to all specified limits in a table, this may be indicated by a caption or by a footnote in the manner indicated in the following examples:

*Example 1.*—Same significant places for all items in a table (illustrating the use of a caption).

	Chemical Composition per cent (to the nearest 0.1 per cent)
Copper.....	4.5 ± 0.5
Iron.....	1.0 max.
Silicon.....	2.5 ± 0.5
Other constituents (magnesium + zinc + manganese).....	0.5 max.
Aluminum.....	remainder

*Example 2.*—Same significant places for all items in a table, with one or two exceptions (illustrating the use of a caption and a footnote).

	Chemical Composition. per cent (to the nearest 0.1 per cent)
Aluminum.....	6.0 ± 0.7
Zinc.....	3.0 ± 0.5
Manganese.....	0.15 min.*
Copper.....	0.05 max.*
Silicon.....	0.5 max.
Other constituents (cadmium + tin + nickel) (nickel 0.03 max.)*.....	0.3 max.
Magnesium.....	remainder

\* To the nearest 0.01 per cent.



*Example 3.*—Significant places not the same for all items in a table (illustrating the use of a footnote).

	Chemical Composition, per cent	
	Min.	Max.
Nickel.....	57	....
Chromium.....	14	18
Manganese.....	....	3
Silicon.....	....	0.40
Carbon.....	....	0.25
Sulfur.....	....	0.03
Iron.....	remainder	

NOTE.—The following applies to all numerical requirements in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained from an analysis should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value.

### Decimals

7. Decimals are preferred to fractions in expressing numerical requirements, where practicable. It is recognized that the use of inches and fractions of inches is standard practice in specifying dimensions for some materials and products.

### Limits

8. (a) Specifications should indicate limits to the range of permissible variations of acceptable material. These may be expressed by the use of maximum and minimum values, or permissible plus and minus deviations from a specified value.

(b) *Double Limits With Preference.*—If there is a preferred value, it should be indicated together with maximum and minimum values, or permissible plus and minus deviations.

#### Example:

Antimony (to the nearest 0.01 per cent) .....  $\left\{ \begin{array}{l} 0.98 \text{ per} \\ \text{cent, pre-} \\ \text{ferred} \end{array} \right\} \left\{ \begin{array}{l} 0.90 \text{ per cent,} \\ \text{min.} \\ 1.10 \text{ per cent,} \\ \text{max.} \end{array} \right.$

or

Antimony (to the nearest 0.01 per cent) .....  $\left\{ \begin{array}{l} 0.98 \text{ per} \\ \text{cent, pre-} \\ \text{ferred} \end{array} \right\} \left\{ \begin{array}{l} -0.08 \text{ per} \\ \text{cent} \\ +0.12 \text{ per} \\ \text{cent} \end{array} \right.$

If the preferred value lies half-way between limits, the maximum deviations permissible may be expressed by the

phrase "plus or minus" or the " $\pm$ " sign. An expression using the phrase "plus or minus" or the sign " $\pm$ " shall be interpreted to indicate a preferred value, and is thus to be considered as having a different meaning from the statement of merely a maximum and a minimum value (see Paragraph (c)).

#### Example:

Antimony (to the nearest 0.01 per cent) .....  $\left\{ \begin{array}{l} 1.00 \pm 0.10 \text{ per} \\ \text{cent} \end{array} \right.$

(c) *Double Limits Without Preference.*—If there is no preference for a specific value, maximum and minimum values alone should be indicated.

#### Example:

Weight (to the nearest 0.01 lb.) .....  $\left\{ \begin{array}{l} 23.40 \text{ lb., min.} \\ 23.50 \text{ lb., max.} \end{array} \right.$

(d) *Single Limit With Preference.*—If the requirements demand a limit in only one direction and there is a preferred value, the preferred value should be indicated together with the maximum or minimum value.

#### Example:

Weight of coating (to the nearest 0.1 mg. per sq. in.) .....  $\left\{ \begin{array}{l} 10.0 \text{ mg. per sq. in.} \\ \text{preferred} \\ 2.0 \text{ mg. per sq. in.,} \\ \text{min.} \end{array} \right.$

(e) *Single Limit Without Preference.*—If the requirements demand a limit in only one direction and there is no preference for a specific value, a maximum or minimum value alone should be indicated.

#### Example:

Weight of coating (to the nearest 0.1 mg. per sq. in.) .....  $\left\{ \begin{array}{l} 2.0 \text{ mg. per} \\ \text{sq. in., min.} \end{array} \right.$

(f) *Percentage Maximum Permissible Deviations.*—If the permissible plus or minus deviation is a direct function of the preferred value, it may be expressed as a percentage, particularly if a wide range of preferred values is expressed or accepted by custom.

### Examples:

The twist, in turns per inch to the nearest turn, shall be as specified, plus or minus 7 per cent.

The resistance in ohms per foot at 20 C. shall be as specified, plus or minus 10 per cent; to the nearest 0.001 ohm for gages Nos. 14 to 21, to the nearest 0.01 ohm for gages Nos. 22 to 31, and to the nearest 0.1 ohm for gages Nos. 32 to 40. On the basis of this specification, the table of minimum and maximum values to be used for purposes of acceptance and rejection would be as follows:

Wire Gage	Resistance,* ohm per foot at 20 C.		
	Min.	Specified	x.
No. 14.....	0.145	0.161	0.177
No. 15.....	0.183	0.203	0.223
.....	.....	.....	.....
No. 21.....	0.732	0.813	0.894
No. 22.....	0.93	1.03	1.13
.....	.....	.....	.....
No. 31.....	7.30	8.33	9.16
No. 32.....	9.3	10.3	11.3
.....	.....	.....	.....
No. 39.....	48.7	54.1	59.5
No. 40.....	56.0	73.3	80.6

\* The following applies to all specified limits in this table: For purposes of acceptance and rejection, an observed value or a calculated value obtained by test should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value.

### Averages

9. (a) Where the term "average" is used without modifying adjectives, it shall be interpreted to indicate the arithmetic mean. If some other average is intended, such as the weighted arithmetic mean, the geometric mean, etc., the standard should so state.

(b) Standards should indicate clearly whether specified limits are to be applied to each single reading, to the average of all readings, to the average of certain subgroups of readings, etc.

(c) If the places of figures to be considered significant in an average differ from those expressed in the specified limit for a single specimen, a statement should be made to this effect.

### Nonnumerical Terms

10. (a) The expressions in the following Paragraphs (b) to (d) are not numerical in character but have a definite relationship to numerical requirements in standards.

(b) *Remainder, Balance, or Difference.*—The analytical method should, in general, include the determination of all the essential ingredients and all of the impurities for which numerical requirements are specified. The term "remainder" (rather than "balance" or "difference") should be used when it is not necessary to make a direct determination of the remaining constituent or constituents involved.

Chemical Composition,  
per cent (to the  
nearest 0.1 per cent)

#### Example:

Copper.....	4.5 ± 0.5
Iron.....	1.0 max.
Silicon.....	2.5 ± 0.5
Other constituents (magnesium + zinc + manganese).....	0.5 max.
Aluminum.....	remainder

(c) *Trace, None, Nil.*—The use of indefinite terms, such as "trace," "none," "nil" should be avoided unless defined as "none by such and such a test."

(d) *Total Impurities.*—The use of indefinite expressions, such as "total impurities," "total other impurities," etc., should be avoided unless the specification likewise lists the elements that should be considered under such expressions.

### Test Method

11. (a) The standard should indicate clearly the test or analytical method to be followed for determining each of the specified numerical requirements. This may be done as follows:

(1) By reference to an A.S.T.M. standard,

(2) By describing the method in detail, or

(3) By stating "any approved method."

(b) Procedures (1) and (2) are recommended as preferable to (3).

#### Permissible Variations for Controlled Constants of Test

12. Permissible variations for controlled constants of testing apparatus and equipment should be designated.

##### Example:

Thickness test— "..... with a load of  $0.100 \pm 0.001$  psi. distributed over a circular area of  $3.00 \pm 0.01$  in. in diameter."

Tensile strength test— ".....with a jaw speed of  $12.00 \pm 0.50$  in. per min."

In the above examples the underlining designates places of figures to be considered significant for purposes of acceptance and rejection of conditions of test.

#### Permissible Variations for Condition of Test Specimens

13. The physical condition of the specimen at the time of test should be specified in all details which influence the characteristics being tested.

##### Examples:

Molded Insulating Materials, Physical Tests.—All specimens shall be conditioned before testing by drying in an oven at  $50 \pm 5$  C. ( $122 \pm 9$  F.) for 48 hr., followed by cooling to room temperature in a desiccator.

Woven Textile Fabrics, Physical Tests.—Standard condition of woven fabrics shall be that reached by the fabric when in moisture equilibrium with a standard atmosphere having a relative humidity of 65 per cent at 70 F. (21 C.). A deviation of plus or minus 2.0 per cent is permitted in relative humidity and plus or minus 2 F. (1.1 C.) in temperature.

#### Sampling and Acceptance Requirements

14. (a) If it is the intent of the specification to provide a definite basis for acceptance and rejection with respect to the numerical requirements, the sam-

pling procedure should be outlined in detail, including:

(1) Method of selecting samples,

(2) Number of tests or analyses, and

(3) Acceptance requirements—including (i) conditions to be met by results of tests if lot is to be considered acceptable, and (ii) conditions for and methods of resampling and retesting.

(b) If there are restrictions as to the number of specimens, tests, or readings to be permitted in obtaining an average for purposes of acceptance and rejection, the standard should so state.

#### Test Readings

15. (a) Where it is important in any operation of a test or an analysis, the specification should indicate how many places of figures the tester or analyst should read on the scale of measurement.

##### Example:

Measure to the nearest 0.001 in.

Read to the nearest 10 lb.

Weigh to the nearest 0.01 g.

Read to the limit of sensitivity of the .....

(b) If the direct reading of a test is taken in the same units of measurement as designated in the specified limit, and if the phrase "to the nearest....." (or underlining) is used in designating the specified limit, this method of expression will indicate to how many places of figures it is *necessary* to read purely for purposes of acceptance and rejection. The practice of reading and recording *additional* places of figures is recommended; among other things, this practice makes available information that may be useful in studies or investigations as to variability of material or of testing methods.

##### Example:

Specified Limit: Weight

(to the nearest 0.1 g.) ..... 5.0 g., min.

For purposes of acceptance and rejection, it would be *necessary* to read the scales of the bal-

ance only to the nearest 0.1 g. If, however, it were subsequently desirable to review past data to study variability, and if the range of variation were only say one or two tenths of a gram, the amount of information available would be meager if values had been recorded only to the nearest tenth of a gram, but would be plentiful if values had been recorded to the nearest hundredth of a gram.

### Calculations

16. (a) The basis of all percentages should be given.

(b) It is recommended that all calculations, such as determinations of tensile strength in pounds per square inch, corrections for temperatures, etc., should carry at least two more places of figures than are to be considered significant for purposes of acceptance and rejection.

*Example.*—The specified tensile strength of a material is 50,000 psi., min. (to the nearest 100 psi.), that is, three places of figures are to be considered significant for purposes of acceptance and rejection. This recommendation calls for carrying out calculations to five places of figures.

If maximum load readings for two test specimens are 5340 and 5080 lb., and if the calculated cross-sectional areas are 0.10240 and 0.10174 sq.

in., respectively, the calculated values of the tensile strength would be as follows:

$$\frac{5340}{0.10240} = 52,148 \text{ psi.}$$

$$\frac{5080}{0.10174} = 49,931 \text{ psi.}$$

Note that if the recommendation in Paragraph (b) had not been followed and if only *one* more place of figures (than are to be considered significant for acceptance and rejection) had been used, the second calculated value would have been  $5080/0.1017 = 49,951$  psi. This value would then have been rounded off to 50,000 and would have been considered *acceptable*, whereas it should have been considered *rejectable*. Using "two more places" in calculations, in accordance with the practice here recommended, usually prevents this type of calculation error from affecting the final value used for purposes of acceptance and rejection.

(c) Digits that are not to be considered in acceptance values should be rounded off (see Section 4 (b)) after all calculations, including averaging, are completed.

*Example.*—Calculated values of Section 16 (b) would be rounded off to give final values of 52,100 and 49,900 psi., respectively, for purposes of acceptance and rejection.

# TENTATIVE DEFINITIONS WITH PROCEDURES RELATING TO CONDITIONING AND WEATHERING<sup>1</sup>



A.S.T.M. Designation: E 41-42 T

ISSUED, 1942.<sup>2</sup>

These Tentative Definitions have been approved by the sponsoring committee and accepted by the Society in accordance with established procedures, for use pending adoption as standard. Suggestions for revisions should be addressed to the Society at 1916 Race St., Philadelphia 3, Pa.

1. These definitions and test procedures pertain to the conditioning and weathering of materials for test purposes. Unless otherwise specified, they are intended to apply to all cases where combinations of atmospheric influences are an essential part of materials testing.

## Definitions

2. (a) *Conditioning*.—The process of subjecting a material to a stipulated influence or combination of influences for a stipulated period of time. The influences may be relative humidity, temperature, light, pressure, air composition, or other factors, used jointly or severally.

NOTE 1.—In some cases a material is subjected to a stipulated influence or combination of influences until substantial equilibrium is reached. Because of the labor involved in determining when equilibrium has been reached, it is customary to subject the material to the stipulated influences for a stipulated period of time, the period of time having been determined with due regard to the nature of the

material, the atmospheric influences, and the conditioning equipment.

NOTE 2.—Conditioning may be used for (a) predicting service behavior, (b) obtaining reproducible results, (c) testing for acceptance, (d) research, and (e) process control; for any single objective or combination of objectives.

NOTE 3.—It is generally true that only a portion of the influences enumerated in the above definition are applicable to any given material. Hence, in any discussion of conditioning, the particular influences that are significant must be stated.

(b) *Average Room Conditions*.—40 per cent relative humidity at a temperature of 77 F. (25 C.).

(c) *Dry Room Conditions*.—15 per cent relative humidity at a temperature of 85 F. (29 C.).

(d) *Moist Room Conditions*.—75 per cent relative humidity at a temperature of 77 F. (25 C.).

(e) *Absolute Humidity*.<sup>3,4</sup>—Concentration (density) of water vapor in the atmosphere expressed as weight per unit volume or weight per unit weight

<sup>1</sup> Under the standardization procedure of the Society, these definitions are under the jurisdiction of the A.S.T.M. Committee E-1 on Methods of Testing.

<sup>2</sup> Accepted by Committee E-10 on Standards, August 24, 1942.

<sup>3</sup> These definitions are identical with those appearing in the Standard Method of Determining Relative Humidity (A.S.T.M. Designation: D 337), 1946 Book of A.S.T.M. Standards, Part III-A.

<sup>4</sup> Editorially revised in 1944.

of (1) dry air or (2) water vapor and air. Grams per cubic foot, grains per pound, or pounds per pound are commonly used.

(f) *Relative Humidity*.<sup>3,4</sup>—Ratio of actual pressure of existing water vapor to maximum possible pressure of water vapor in the atmosphere at the same temperature, expressed as a percentage.

(g) *Dew Point*.<sup>3</sup>—Temperature at which condensations of moisture would occur if the existing atmosphere were cooled without change in vapor pressure.

(h) *Vapor Pressure*.<sup>3</sup>—Component of atmospheric pressure caused by presence of water vapor, usually expressed in inches of mercury.

(i) *Dry Bulb Temperature*.<sup>3</sup>—Temperature of the air as indicated by a standard thermometer.

(j) *Wet Bulb Temperature*.<sup>3</sup>—For purposes of this method, temperature indicated by the wet bulb thermometer of a standard sling psychrometer or its equivalent. Theoretically, temperature at which the atmosphere would become saturated by evaporation of water without loss or gain in total heat content of the air and vapor.

(k) *Hygrometer*.<sup>3</sup>—Any properly calibrated instrument which indicates directly or indirectly the humidity of the air.

(l) *Absorption Hygrometer*.<sup>3</sup>—Any one of several types of hygrometers containing a hygroscopic substance the length, thickness, or weight of which is a measurable index of the humidity of the atmosphere. Usually such devices are graduated directly either in relative humidity or in regain of stock in process, and are continuously indicating.

(m) *Wet and Dry Bulb Hygrometer or Psychrometer*.<sup>3</sup>—Contains two matched thermometers, one plain and one having a wet bulb covering as hereinafter specified—for simultaneously indicating the

dry and wet bulb temperatures of the atmosphere.

(n) *Recording Hygrometer or Psychrometer*.<sup>3</sup>—Any approved type of hygrometer or psychrometer equipped with automatic clocking and recording device capable of making a continuous time - relative humidity autograph, or time - wet and dry bulb temperature autograph.

(o) *Sling Psychrometer*.<sup>3</sup>—A psychrometer containing matched dry and wet bulb thermometers suitably mounted for swinging through the atmosphere, for simultaneously indicating dry and wet bulb temperatures. The thermometers shall be so mounted as to act independently and to face the air current during swinging.

#### Relative Humidity Determination

3. (a) Relative humidity shall be determined in accordance with the Standard Method of Determining Relative Humidity (A.S.T.M. Designation: D 337) of the American Society for Testing Materials.<sup>5</sup>

(b) For determining relative humidity from psychrometric observations within the normal temperature range 0 to 112 F. (-20 to +45 C.), reference may for convenience be made to the Psychrometric Table for Relative Humidity.<sup>6</sup>

#### Room Temperature Determination

4. Whenever the room temperature is to be determined in any test method the following procedures shall be used:

(a) All air temperatures shall be measured with an A.S.T.M. Gravity Thermometer 12F - 39 or 12C 39 having a range of -5 to +215 F. or -20 to +102 C., as specified, and conforming to the requirements of the

<sup>3</sup> 1946 Book of A.S.T.M. Standards, Part III-A.

<sup>4</sup> *Proceedings*, Am. Soc. Testing Mats., Vol. 31, Part 1, p. 567 (1931); also in "A.S.T.M. Standards on Textile Materials," October, 1946, p. 416, available as a separate publication.

Standard Specifications for A.S.T.M. Thermometers (A.S.T.M. Designation: E 1) of the American Society for Testing Materials.<sup>7</sup>

(b) All air temperature (room temperature) measurements shall be made with the thermometer located within 2 ft. in a horizontal direction of the point at which the test is actually being made. In case the test involves any heat or heating apparatus, the thermometer

shall be shielded from direct radiation as well as convection currents.

#### Standard Laboratory Atmosphere

5. Whenever the materials to be tested are known to be sensitive to variations in temperature or moisture, or both, the tests shall be conducted in a room or chamber of controlled humidity and temperature. Unless otherwise specified, the tests shall be made in the standard laboratory atmosphere having a relative humidity of  $50 \pm 2$  per cent at a temperature of  $77 \pm 2$  F. ( $25 \pm 1.1$  C.).

<sup>7</sup> Appears in this publication, see Contents in Numeric Sequence of A.S.T.M. Designations at front of book.



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TENTATIVE REVISIONS  
OF  
A.S.T.M. STANDARDS ON NONMETALLIC  
MATERIALS—ELECTRICAL INSULATION,  
PLASTICS, RUBBER, PAPER,  
SHIPPING CONTAINERS, ADHESIVES

*As of December, 1946*

(For change in status of any of these tentative revisions, see supplementary publications to be  
issued in 1947 and 1948.)

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Tentative revisions of A.S.T.M. Standards are printed for one or more years with a view of eliciting criticisms, of which the committee concerned will take due cognizance before recommending final action.

New Tentative Revisions and any changes in these Tentative Revisions, issued in 1947 and 1948, will appear in the 1947 and 1948 Supplements to the Book of A.S.T.M. Standards. It is important that these Supplements be consulted in case any change of status has occurred with respect to any of the Tentative Revisions.

Members of the Society and others are invited to direct written criticisms of any of these Tentative Revisions to the Headquarters of the Society, 1916 Race St., Philadelphia 3, Pa.

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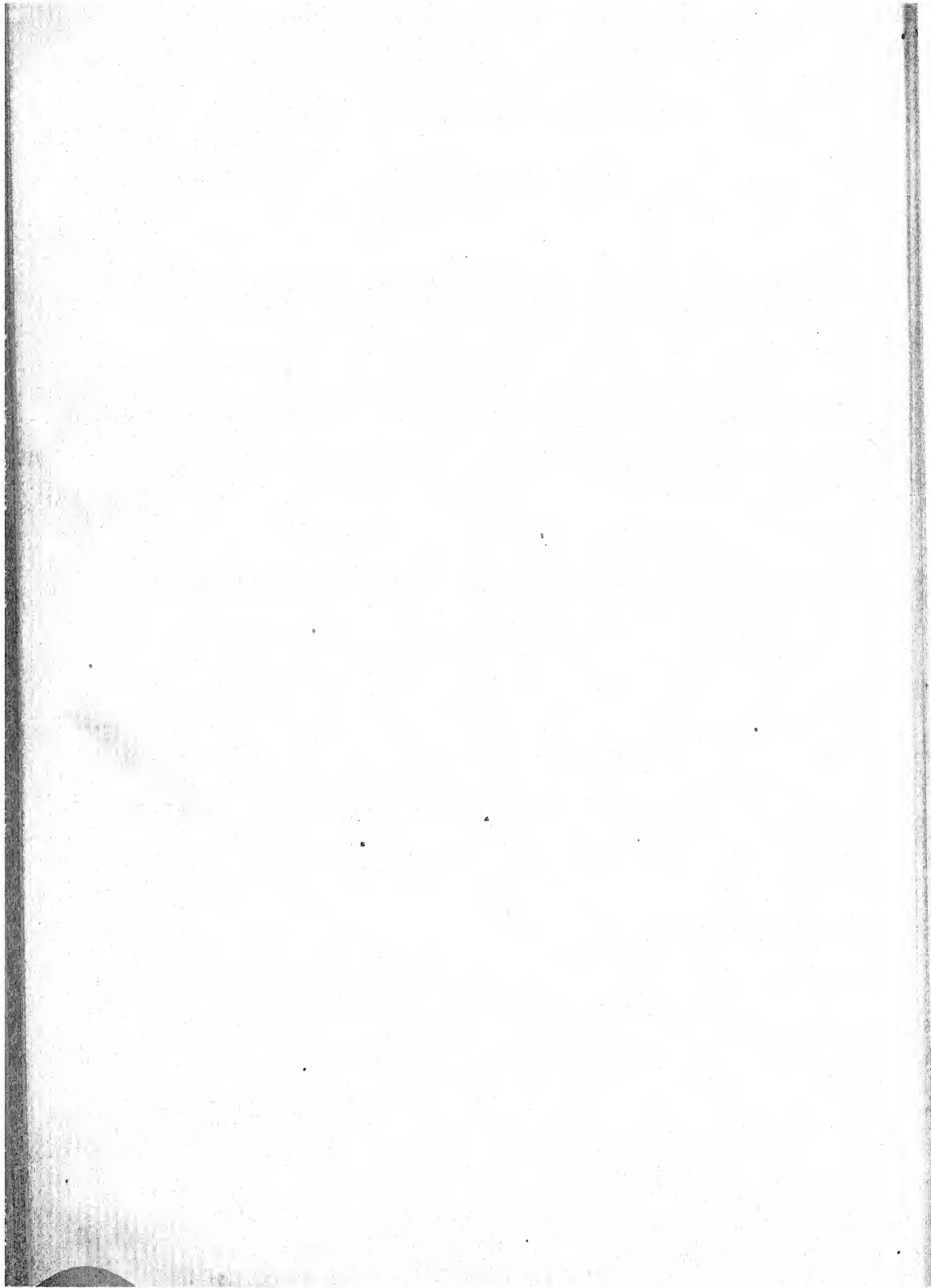
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<sup>1</sup> See Supplements to be issued in 1947 and 1948 for revisions or change in status of any of these tentative revisions.



# TENTATIVE REVISIONS OF A.S.T.M. STANDARDS RELATING TO NONMETALLIC MATERIALS—ELECTRICAL INSULATION, PLASTICS, RUBBER, PAPER, SHIPPING CONTAINERS, ADHESIVES

*As of December, 1946*

## Standard Methods of Testing Laminated Round Rods Used in Electrical In- sulation (D 349 - 46):<sup>1</sup>

*Section 4.*—Change this section to read as follows by the addition of the italicized words and the omission of those in brackets:

4. The test specimens shall be about 12 in. in length. When the diameter of the rod is  $\frac{1}{4}$  in. or over, a groove [1 in. in width] *2.25 in. in length*, and  $\frac{1}{32}$  in. in depth with corners rounded to a radius of 3 in. shall be machined around the rod at the center.

*Tentative revision, submitted June, 1944.*

## Standard Methods of Testing Sheet and Plate Materials Used in Electrical Insulation (D 229 - 46):<sup>2</sup>

*Sections 10 to 13.*—In place of the test for flexural strength described in these sections substitute the following:

### FLEXURAL STRENGTH

*For Materials  $\frac{1}{8}$  in. or Over in Thickness (Note)*

10. (a) Except for definitions of cutting directions of specimens as specified in Section 2 and for conditioning as specified in Section 3, the flexural strength properties including modulus of rupture and modulus of elasticity in vending shall be determined in accordance with the Tentative Method of Flexural Test of Plastics (A.S.T.M. Designation: D 790).<sup>3</sup>

(b) Five specimens cut lengthwise and five specimens cut crosswise of the sheet shall be tested for flatwise test. Where edgewise tests are desired, five specimens shall also be cut lengthwise and five crosswise of the sheet.

NOTE.—Conventional flexural tests in a flatwise direction are not recommended for materials thinner than  $\frac{1}{32}$  in. nor in an edgewise direction for materials thinner than  $\frac{1}{16}$  in. It should also be noted for the thin sections, loads required

are so small that special low load testing machines may be required to maintain the limits of accuracy specified.

*Tentative revision, submitted December, 1944.*

*Sections 39 to 42.*—Replace the test for Rockwell hardness in these sections by the following:

39. Rockwell hardness of all sheet and plate insulating materials shall be determined in accordance with the Tentative Method of Test for Rockwell Hardness of Plastics and Electrical Insulating Materials (A.S.T.M. Designation: D 785).<sup>4</sup>

*Tentative revision, submitted June, 1944.*

## Standard Methods of Testing Laminated Tubes Used in Electrical Insulation (D 348 - 46):<sup>5</sup>

*Sections 3 and 4.*—Rename as Sections 4 and 5, and revise the description of the apparatus and test specimen for the tension test to read as follows:

4. *Apparatus.*—Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied. Jaws which tighten under load, such as wedge-grip jaws, shall be used with the specimen properly aligned.

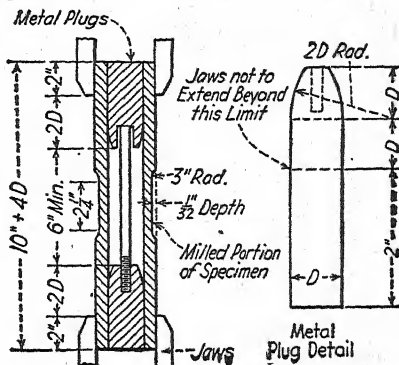


FIG. 1.—Assembly for Tension Test of Tubes.

<sup>1</sup> See p. 31.

<sup>2</sup> See p. 36.

<sup>3</sup> See p. 858.

<sup>4</sup> See p. 868.

<sup>5</sup> See p. 52.

5. *Test Specimens*.—The length of the test specimens shall be 10 in. plus 4 times the inside diameter of the tube. When the wall thickness of the tubing is  $\frac{1}{8}$  in. or over, a groove  $2\frac{1}{2}$  in. in length in the flat portion and  $\frac{1}{16}$  in. in depth shall be machined around the tube at the center with corners rounded to a radius of 3 in. Steel or brass plugs, whose length is 2 in. plus twice the diameter, and which conform to Fig. 1 shall be used to plug the ends of the specimen to prevent crushing. The plugs shall fit the specimen within 0.010 in. They may be located conveniently in the tube by separating and supporting them on a metal rod the lower end of which is screwed into the lower plug and the upper end of which rests in a loose socket as shown.

*Tentative revision, submitted June, 1944.*

#### Standard Methods of Testing Flexible Varnished Tubing Used for Electrical Insulation (D 350 - 45):<sup>6</sup>

*Title*.—Change the term “varnished tubing” to read “treated sleeving” in the title and wherever it occurs throughout the text of these methods.

*Section 1*.—Delete the words “and saturated sleeving.”

*Tentative revision, submitted December, 1944.*

*Note*.—Change the note under Section 1 from its present form: namely,

NOTE.—The term “varnished tubing” as used in these methods refers to braided cotton sleeving coated, or impregnated and coated, with varnish, lacquer, or a combination of lacquer and varnish, or similar coatings. The term “saturated sleeving” refers to braided cotton sleeving impregnated with varnish but the coating need not be continuous as it is for varnished tubing.

to read as follows:

NOTE.—The term “treated sleeving” as used in these methods refers to cotton, fiberglass, or rayon braided sleeving impregnated, coated, or impregnated and coated with an oil varnish, lacquer or a combination of lacquer and oil varnish.

*Tentative revision, submitted December, 1944.*

*Table I*.—Delete from Table I the requirements for size No. 19 and add a new size No. 24 with the following requirements as to inside diameter: 0.027

in. max., 0.020 in. min., 0.022 in. nominal.

Insert in the table the following nominal inside diameters for the sizes indicated:

Size	Inside Diameter, in. Nominal
1.....	1.018
7/8.....	0.893
5/8.....	0.640
1/2.....	0.512
7/16.....	0.450
3/8.....	0.387
3/4.....	0.768

*Tentative revision, submitted December, 1944.*

*Section 13*.—Under aging test add the following as a new Section, renumbering the remaining sections accordingly: “13. This test shall be applicable to treated sleeving as defined in Section 1, except that it shall not apply to fiber glass.”

*Tentative revision, submitted December, 1944.*

*Section 14. (a)*.—In the fourth line of the table for diameter of test rod, change “Nos. 6 to 11, incl.” to read “No. 6 and smaller.” Delete the last line which reads “No. 12 and smaller..... $\frac{1}{8}$ .”

*Tentative revision, submitted December, 1944.*

#### Standard Methods of Testing Electrical Insulating Oils (D 117 - 43):<sup>7</sup>

The Tentative Methods of Testing Electrical Insulating Oils (D 117 - 46 T)<sup>8</sup> are intended to replace, when adopted, the present Standard Methods D 117 - 43.

*Tentative revision, submitted November, 1946.*

#### Standard Specifications for A.S.T.M. Thermometers (E 1 - 46):<sup>9</sup>

The Tentative Specifications for A.S.T.M. Thermometers (E 1 - 46 T)<sup>10</sup> are intended to be added, when adopted, to the present Standard Specifications E 1 - 46.

*Tentative revision, submitted December, 1946.*

<sup>7</sup> See p. 65.

<sup>8</sup> See p. 600.

<sup>9</sup> See p. 454.

<sup>10</sup> See p. 1281.

<sup>6</sup> See p. 61.

# INDEX OF

## BOOK OF A.S.T.M. STANDARDS, PART III-B

### STANDARDS AND TENTATIVES ON NONMETALLIC MATERIALS— ELECTRICAL INSULATION, PLASTICS, RUBBER, PAPER, SHIPPING CONTAINERS, ADHESIVES

This Index covers solely those standards and tentatives appearing in this volume. A Combined Index, covering all standards and tentatives appearing in all five Parts of the Book of A.S.T.M. Standards, is furnished separately. The Combined Index will be revised annually to incorporate the latest references to the standards and tentatives, whether they appear in the Book of Standards or in the 1947 and 1948 Supplements thereto. The Combined Index will be particularly helpful after one of the Supplements has been issued.

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